

# Microencapsulation of Oils: A Comprehensive Review of Benefits, Techniques, and Applications

Amr M. Bakry, Shabbar Abbas, Barkat Ali, Hamid Majeed, Mohamed Y. Abouelwafa, Ahmed Mousa, and Li Liang

**Abstract:** Microencapsulation is a process of building a functional barrier between the core and wall material to avoid chemical and physical reactions and to maintain the biological, functional, and physicochemical properties of core materials. Microencapsulation of marine, vegetable, and essential oils has been conducted and commercialized by employing different methods including emulsification, spray-drying, coaxial electrospray system, freeze-drying, coacervation, *in situ* polymerization, melt-extrusion, supercritical fluid technology, and fluidized-bed-coating. Spray-drying and coacervation are the most commonly used techniques for the microencapsulation of oils. The choice of an appropriate microencapsulation technique and wall material depends upon the end use of the product and the processing conditions involved. Microencapsulation has the ability to enhance the oxidative stability, thermostability, shelf-life, and biological activity of oils. In addition, it can also be helpful in controlling the volatility and release properties of essential oils. Microencapsulated marine, vegetable, and essential oils have found broad applications in various fields. This review describes the recognized benefits and functional properties of various oils, microencapsulation techniques, and application of encapsulated oils in various food, pharmaceutical, and even textile products. Moreover, this review may provide information to researchers working in the field of food, pharmacy, agronomy, engineering, and nutrition who are interested in microencapsulation of oils.

Keywords: essential oil, marine oil, vegetable oil, microencapsulation

# Introduction

Marine, vegetable, and essential oils, and their components, are gaining increasing interest in the food, agriculture, pesticide, textile, cosmetic, and pharmaceutical industries because of their natural and safe status, wide acceptance by consumers, and multidimensional functional properties. Marine oils are in high demand because they contain large amounts of omega-3 polyunsaturated fatty acids ( $\omega$ -3 PUFAs), which have numerous health benefits. Vegetable oils also offer several health benefits compared to animal oils, including butter and ghee, when consumed by humans. Essential oils obtained from plants are complex mixtures of natural volatile compounds. They give plants their characteristic odors and are a common source of bioactive ingredients. In addition to basic nutrition, they are consumed for their various functional properties including uses such as gastronomic, nutritional, organoleptic, antioxidant, anti-inflammatory, antivasocon-

strictive, anti-arrhythmic, antithrombotic, antimicrobial, antihypertension, antiulcer, anti-aging, anticancer, antidiabetic, antidepressant, antipyretic antitussive, antidandruff, antinociceptive, and insect repellent (Satchell and others 2002; Zhao and others 2004; Deng and others 2005; Nadig and Laxmi 2005; Williams and others 2007; Mandal and Mandal 2011; Chung and others 2013; Ozyildiz and others 2013; Che Idris and others 2014; Ito and others 2014; Jeena and others 2014; Jouki and others 2014a; Lockyer and Rowland 2014; Olmedo and others 2014; Szabo and others 2014). Marine, vegetable, and essential oils have recently gained a great popularity and scientific interest. Although several attempts have been made to harness the full potential of these oils, they are chemically unstable and susceptible to oxidative deterioration and loss of volatile compounds, especially when exposed to oxygen, light, moisture, and heat. The quality of a product fortified with the oils may deteriorate due to oxidative degradation, formation of unpleasant tastes and off-flavors, and the generation of free radicals. These changes have a negative effect on the shelf-stability, sensory properties, and overall acceptability of the developed products (Velasco and others 2003). To develop natural health products containing marine, vegetable, or essential oils, microencapsulation technology could be a viable option to maintain their biological and functional characteristics.

Microencapsulation is a method in which tiny particles or droplets are surrounded by a coating wall, or are embedded in a homogeneous or heterogeneous matrix, to form small capsules (Gharsallaoui and others 2007; Calvo and others 2011). It can

MS 20151241 Submitted 30/7/2015, Accepted 17/9/2015. Authors Bakry, Abbas, Ali, Majeed, Abouelwafa, Mousa, and Liang are with the State Key Laboratory of Food Science and Technology, School of Food Science and Technology, Jiangnan Uniw, Wuxi, Jiangsu, 214122, PR China. Authors Bakry and Abouelwafa are with the Dept. of Dairy Science, Faculty of Agriculture, Suez Canal Uniw, Ismailia, 41522, Egypt. Author Mousa is with the Dept. of Dairy Science, Faculty of Enwironmental Agricultural Science, Suez Canal Uniw, 45516, El Arish, Egypt. Author Abbas is with the Dept. of Biosciences, COMSATS Inst. of Information Technology, Park Road, Islamabad, 45550, Pakistan. Direct inquiries to author Liang (E-mail: liliang@jiangnan.edu.cn).

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Figure 1-Composition of an oil microcapsule in simplified form.

envelop a solid, liquid, or gaseous substance within another substance in a very small sealed capsule. The core material is gradually diffused through the capsule walls, thereby offering controlled release properties under desired conditions (Fang and Bhandari 2010). Therefore, microencapsulation technology can be used to deliver bioactive components, improving their handling properties. Fox example, physical encapsulation of sensitive oils in small capsules could prevent oxidation triggered by moisture, metal ions, oxygen, and heat (Gharsallaoui and others 2007; Calvo and others 2011). Briefly, microencapsulation can be defined as a process of building a functional barrier between the core and the wall material to avoid chemical and physical reactions and to maintain the biological, functional, and physicochemical properties of the core materials.

The schematic diagram of microcapsules showing core and wall material is shown in Figure 1. Generally, microcapsules consist of a core material, which is referred to as the internal phase or fill, and a wall referred to as the coating, shell, or membrane. Wall material determines the stability of microparticles, the process efficiency, and the degree of protection for the core. Wall materials used commonly for the microencapsulation of oils include synthetic polymers and natural biomaterials (usually carbohydrates and proteins). Some of microcapsule models (Raybaudi-Massilia and Mosqueda-Melgar 2012) are shown in Figure 2. Generally, size and shape of the formed microcapsules depend on wall materials and the methods used to prepare them. Commonly used microencapsulation techniques are: emulsification, spray-drying, coaxial electrospray system, freeze-drying, coacervation, in situ polymerization, extrusion, fluidized-bed-coating, and supercritical fluid technology (Devi and Maji 2009; Anwar and others 2010; Quispe-Condori and others 2011; Sanchez-Navarro and others 2011; Liu and others 2012; Almeida and others 2013; Soliman and others 2013; Botrel and others 2014a,b; Sutaphanit and Chitprasert 2014; Tatar and others 2014; Wang and others 2014a). In addition, wall composition and microencapsulation techniques may also determine functional properties and potential applications of encapsulated components.

Controlled release has been defined according to Pothakamury and Barbosa-Cánovas (1995) as a method by which one or more active agents or ingredients are made available at a desired site and time at a specific rate. Controlled release technology is used to deliver various compounds such as drugs, pesticides, fragrances, or flavors at recommended rates, together with improved efficacy



Figure 2–Different types of microcapsules: (i) simple microcapsule, (ii) matrix (microsphere), (iii) irregular microcapsule, (iv) multicore microcapsule, (v) multiwall microcapsule, and (vi) assembly of microcapsule.



Figure 3–Schematic diagram of oil controlled release mechanism.

and safety (Martins and others 2014a). In short, Figure 3 shows the schematic representation of oil controlled release mechanism.

Obviously, there are some reviews about microencapsulation of  $\omega$ -3-rich oils regarding the progress in enzymatic concentration (Kralovec and others 2012) and a patent-based review on industrial approaches (Drusch and Mannino 2009). Moreover, microencapsulation of essential oils regarding properties, applications, and biodegradable polymers in the cosmetic field, focusing the use of polylactide as the base material to encapsulate thyme oil by the coacervation technique (Martins and others 2014a). In this review, we will shed light on the benefits inherent to marine, vegetable, and essential oils and role of microencapsulation to further enhance functional properties of encapsulated bioactives. Additionally, we illustrate the microencapsulation techniques of oils. Moreover, we mention and update the applications of encapsulated oils in various food, pharmaceutical, and textile products.

# Benefits of Oils and Their Encapsulation Marine oils

Fish oil. Fish oils contain essential PUFAs, including  $\omega$ -3 PUFAs eicosapentaenoic acid (EPA) and docosahexaenoic acid

(DHA) and  $\omega$ -6 PUFAs linoleic (C18:2) and arachidonic (C20:4) acids). The long-chain  $\omega$ -3 PUFAs, found in abundance in fish oil, are well documented for a range of health benefits (McLennan and Abeywardena 2005; Weitz and others 2010). They play a possible role in fetal and early childhood development and improved cognition. Long-chain ω-3 PUFAs have anti-inflammatory effects and provide protection against various cancers in human cell lines (Wendel and Heller 2009) and have a potential to improve cardiovascular and mental health (Ruxton and others 2007). In clinical studies, it has been shown that  $\omega$ -3 fatty acids from fish oil can improve brain function and help to prevent diseases including immune response disorders, ulcerative colitis, and Crohn's disease (Jordan 2010; Eckert and others 2010). Recently, it has been found that fish oil serves to treat memory deficit (Ferreira and others 2014). Lauterbach and Pawlik (2014) demonstrated that DHA is a major structural lipid in sensory and vascular retina tissue and its deficiency may trigger the structural and functional abnormalities in the visual system. It was reported that the fish oil is a perfect dietary supplement for growing rabbits as no adverse effects were detected on the histological structure of liver and kidney (El-Moghazy and others 2014).

The major hurdles associated with the incorporation of fish oils into food products are insolubility in water, susceptibility of PUFAs to oxidative deterioration, and development of undesirable rancid odor and taste (Augustin and others 2006). Utilization of fish oil in aqueous food systems can be improved by using encapsulation technology (Patrick and others 2013). By using the complex coacervation method, gelatin and sodium carboxymethylcellulose (NaCMC) used as wall material could protect EPA and DHA in fish oil (Patrick and others 2013). Tuna oil microencapsulated in gelatin-sodium hexametaphosphate complex particles showed greater stability than nonencapsulated oil (Wang and others 2014a). Barley protein-based microcapsules possessed a strong ability to protect fish oil against oxidation, and make them suitable for use in liquid/semiliquid food systems (Wang and others 2011). A stable emulsion containing menhaden oil was produced by using sodium caseinate (SC) and soluble rice bran fiber (SRBF), and SRBF provided better oxidative stability during storage and spray-drying (Wan and others 2011a). Augustin and others (2011) demonstrated that microencapsulation of fish oil, tributyrin, and resveratrol in oil-in-water emulsions, stabilized by a heated mixture of caseinate, glucose, and modified resistant starch, increased the levels of radioactivity from the bioactives in the blood and liver, thereby increasing their bioavailability.

Microalgal oil. Microalgal are photosynthetic microorganisms, which can produce large amounts of lipids containing high-value bioactives. The lipid contents in microalgae range from 15% to 77%, depending on the species (Table 1) or culture conditions (Chisti 2008; Rawat and others 2013). Microalgal oil contains considerable amounts of PUFAs, especially DHA, and can be used as a supplement (Sijtsma and de Swaaf 2004; Klok and others 2014). Most microalgal oils are a good source of  $\omega$ -3 PUFAs and serve as an alternative to fish oil; in addition, they contain sterols and carotenoids. The intake of carotenoids is, however, definitely significant and could give the microalgal oils a nutritional added value compared to fish oil (Ryckebosch and others 2014). Recently, it has been found that microalgal oil may offer potent protective effects against chronic diseases, such as cardiovascular disease and stroke (Szabo and others 2014). When microalgal oil was encapsulated in gelatin-gum arabic (GA) complex-coacervated matrices by transglutaminase (TG) as cross-linking agent, the oil release could be regulated by changing the cross-linking parameters, with

the optimum ones as follows: hardening for 6 h at 15 °C and pH 6.0 with a TG concentration of 15 U/g gelatin (Zhang and others 2012a). Bao and others (2011) demonstrated that the powder of microalgal oil enhanced its physicochemical properties and oxidative stability when prepared with SC cross-linked by microbial TG and maltodextrin (MD) by spray-drying.

# Vegetable oils

**Linseed oil.** Linseed oil (flaxseed oil) is a good source of  $\omega$ -3 PUFAs (Rubilar and others 2012a), which contain more than 50%  $\alpha$ -linolenic acid (ALA) also and high contents of monounsaturated oleic acid (21.2%) and linoleic acid (LA, 13.96%). The oil also contains about 79 mg/100 g vitamin E (tocopherols), in which  $\gamma$ tocopherol is dominant and acts as an essential nutrient (Bozan and Temelli 2008), functioning mainly as a chain-breaking antioxidant that protects cell membranes against oxidative damage. Flaxseed oil thus acts as an essential nutrient. Encapsulation of flaxseed oil has been studied using MD combined with other materials. In the case of GA, whey protein concentrate (WPC), modified starch (MS) Hi-Cap 100<sup>TM</sup>, and Capsul TA<sup>®</sup>, it was found that the best encapsulation efficiency was obtained for MD:Hi-Cap combination followed by MD:Capsul combination, while the lowest encapsulation efficiency was for MD:WPC combination, which provided the best protection against lipid oxidation (Carneiro and others 2013). It has been observed that flaxseed oil could be entrapped efficiently with MD combined with chickpea protein isolate or lentil protein isolate by spray-drying and freeze-drying, providing a protective effect against oxidation over a storage period of 25 d at room temperature and delivering more than 80% of the encapsulated oil to the gastrointestinal tract (Karaca and others 2013a,b). By encapsulating linseed oil using GA or its mixture with MD as wall materials, a soup powder enriched with  $\omega$ -3 fatty acid was prepared that was acceptable to consumers (Rubilar and others 2012a). Microcapsules containg linseed oil made of GA presented more than 90% encapsulation efficiency and provided protection from oxygen and high temperature. Addition of the microcapsules did not change the appearance of fortified bread but, ALA conent was reduced significantly during bread manufacturing (Gallardo and others 2013)

Soybean oil. Due to its oil-rich seeds, the soybean is the most important legume crop in the world. Soybean oil is classified as polyunsaturated oil, which includes about 15% saturates, 24% monounsaturates, and 61% polyunsaturates of which the contents of LA and linolenic acid are, respectively, about 53.2% and 7.8%. Soybean oil is also an important source of vitamin E. It helps to lower the serum cholesterol and low-density lipoprotein (LDL) levels and to prevent atherosclerosis and heart disease. It can also be used as a potential hypocholesterolemic agent (Kummerow and others 2007; Gresshoff 2013). Spray-dried soybean oil emulsions were prepared using SC or whey protein; the encapsulation ability of the latter is rather low compared with the former. The emulsion droplet structure, the release of the oil onto the powder surface, and the size of the emulsion droplets after redispersion were affected by the addition of lactose (Faldt and Bergenstahl 1996a,b). When encapsulating soybean oil using legume proteins as wall materials, it was found that microencapsulation properties of soy protein isolate (SPI) are considerably better than kidney, red, and mung bean protein isolates, in terms of the oil retention efficiency (RE), redispersion and dissolution behavior, and storage stability at high humidity of spray-dried powders, except RE for kidney bean protein isolate (Liu and others 2014).

Oil type	Species	Source of oil extraction	Major components	References
Microalgal oil	Phaeodactylum tricornutum, Rhodomonas salina, Tetraselmis suecica, Thalassiosira pseudorana, Nannochloropsis gadirana, Nannochloropsis oculata, Porphyridium cruentum, &	Microplants or photosynthetic microorganisms	DHA, DPA, & hexadecanoic acid	Ryckebosch and others (2014); Klok and others (2014); Bao and others (2011)
Linseed oil Soybean oil Evening primrose oil Sunflower oil Kenaf seed oil	Linum usitatissimum Glycine max Oenothera biennis Helianthus annuus Hibiscus cannabinus	Seeds Seeds Seeds Seeds Seeds	α-Linolenic (ALA), oleic, & linoleic acids Linoleic, linolenic, & oleic acids γ-Linolenic acid & linoleic acid Linoleic, oleic & stearic acids Linoleic, oleic, palmitic, & ALA acids	Bozan and Temelli (2008); Karaca and others (2013b) Gresshoff (2013) Montserrat-de la Paz and others (2014) Pal (2011) Ng and others (2013a); Ng and others (2013b);
Red pepper seed oil Jojoba oil Walnut oil	Capsicum annuum Simmondsia chinensis Juglans regia	Seeds Seeds & fruits	Linoleic, oleic, hexadecanoic, stearic, & linolenic acids Eicosenoic, docosenoic, & oleic acids Linoleic, oleic, linolenic, palmitic, & stearic acids	Nazimkinal and others (2013) Ozyildiz and others (2013); Wang and others (2014b) Abdel-Mageed and others (2011); Calvo and others (2011); Pereira and others (2008); Li and others (2003)
Macadamia oil Olive oil	Macadamia integrifolia Olea europaea	Fruits Whole fruit	Oleic, palmitoleic, linoleic, & $\alpha$ -linolenic acids Oleic, linoleic, & palmitic acids	Irene and Manohar (2008) Lockyer and Rowland (2014): Koutsopoulos and others (2008)
Red palm oil Sea buckthorn oil	Elaeis guineensis Hippophae rhamnoides	Palm fruit Fruit flesh & peels	Palmitic, oleic, linoleic, stearic, & myristic acids Linoleic, ALA, oleic, palmitic, stearic, & vaccenic acids	Mukherjee and Mitra (2009) Hukherjee and Mitra (2009) Ito, and others (2014); Yang and Kallio (2001); Basu
Avocado oil Wheat germ oil	Persea americana Triticum vulgare	Fruit flesh Wheat germ	Oleic, palmitic, linoleic, & palmitoleic Linoleic, palmitic, oleic, & linolenic acids	Requejo and others (2004), Amig and others (2004) Can and others (2000); Ozdemir and Topuz (2004) (2003): Gabors and others (2000): Megahad and El Kinawy
Orange peel oil	Citrus sinensis "sweet" & Citrus aurantium "bitter"	Fruit peel	Limonene, $\alpha$ -pinene, sabinene, myrcene, linalool, $\alpha$ -terpineol, citronellal	Frerot and Decorgant (2004); Viuda-Martos and others (2008)
Mandarin peel oil	Citrus reticulata	Fruit peels & leaves or petit grain	Limonene, limonene/y-terpinene and linalyl acetate/limonene "peels" & sabinene/linalool, y-terpinene/linalool and "lastice or natit creain"	Li and others (2006); Lota and others (2001)
Kaffir lime oil	Citrus hydrix	Fruit peels & leaves	$\beta$ -prince & limonene "peels" & (-)-(5)-citronellal "lanee"	Adamiec and others (2012)
Oregano oil Cardamom oil	Origanum vulgare Elettaria cardamomum	Leaves Seeds	Carverol, thymol, & p-cymene α-terpineol, myrcene, limonene, menthone, β- mellandrene 1 8-cineol	Olmedo and others (2014) Sengottuvelu (2011); Kumari and Dutta (2014); Berictain and others (2001)
Mint oil	Mentha piperita	Leaves	Menthol, menthone, menthyl acetate, menthofuran, & 1,8-cineol	Is can and others (2002); CU01, Sumar and others (2004); Sarkar and others (2003); Sarkar and others (2012); Hwang and others (2006); Baranauskiene and others (2007)
Holy basil oil	Ocimum sanctum	Leaves	Methyl eugenol, caryophyllene, & eugenol	Nadig and Laxmi (2005); Baliga and others (2013); Kumar and others (2010); Singh and others (2007): Khanar and others (2010)
Rosemary oil	Rosmarinus officinalis	Flowers & leaves	( $lpha$ & $eta$ -Pinene), camphene, limonene, & camphor	(2007a), Niamina and Others (2010) Rozman and Jersek (2009) de Barros Fernandes and
Pterodon oil	Pterodon emarginatus	Fruits	$eta$ -caryophyllene, $\delta$ -elemene, $lpha$ -copaene, $eta$ -cubebene, allo- aromadendrene, $lpha$ - cubebene, $\&$ $\gamma$ -muurulene	Evangelista and others (2007); Dutra and others (2009), Barbosa Raposo and others (2011); Alves
Garden cress oil	Lepidium sativum	Seeds	ALA, oleic, linoleic, eicosanoic, palmitic, erucic, arachidic, & stearic acids	Umesha and others (2015); Moser and others (2009)
				(Continued)

Table 1-Commonly used oils, their source, extraction, and their major components

lable 1–Continued.				
Oil type	Species	Source of oil extraction	Major components	References
Coffee oil	Coffea arabica & Coffea canephora	Beans	Linoleic, palmitic, stearic, & oleic acids	Wagemaker and others (2011); Carvalho and others
Lippia oil	Lippia sidoides	Leaves	Thymol, carvacrol, estragole, linalool, & limonene	Costa and others (2001); Pascual and others (2001); Botelho and others (2007); de Lima and others (2013); Camurca-Vasconcelos and others (2007); Cavalcanti and others (2010); Fernandes and others (2008)
Lemongrass oil	Cymbopogon citratus	Leaves	Citral, geraniol, myrcene, limonene, citronella, borneol, nervi acetate. Z-carvobivllene	Kpoviesi and others (2014); Naik and others (2010); Costa and others (2011)
Lemon myrtle oil	Backhousia citriodora	Leaves	Neral & geranial	Forbes-Smith and Paton (2002); Southwell and others (2000)
Pitaya seed oil	Hylocereus polyrhizus	Seeds	Linoleic, oleic, palmitic, stearic, vaccenic & linolenic acids	Limento others (2012); Hor and others (2012); Lim and others (2010); Tenore and others (2012)
Cinnamon leaf oil	Cinnamomum zeylanicum	Leaves	Eugenol, cinnamaldehyde	Ayala-Zayala and others (2008); Singh and others (2007b)
Garlic oil	Allium sativum	Cloves	Diallyl disulfide & diallyl trisulfide	Ayala-Zavala and others (2008); Rees and others (1933): Jawrence and Jawrence (2011)
Tea tree oil	Melaleuca alternifolia	Leaves	Terpinen-4-ol, $\gamma$ -terpinene, $\alpha$ -terpinene, p-cymene, terpinolene, $\alpha$ -terpineol, $\alpha$ -pinene, limonene, $\aleph$ 1.8-cineole	Thomsen and others (2013)
Thyme oil	Thymus vulgaris	Flowers & leaves	Thymol, carvacrol, cinnamaldehyde, geraniol, eugenol, n-rymene myrrene hyrneol & linalool	Sipailiene and others (2006); Navarrete and others (2010)
Caraway oil Vetiver oil	Carum carvi Chrysopogon zizanioides & Vetiveria	Seeds Roots	Petroselinic, linoleic, myristic, palmtic, & linolenic Khusimol, $\alpha$ -vetivone, $\beta$ -vetivone, khusimone, zizaene,	Laribi and others (2009); Bylaite and others (2001) Danh and others (2009)
Camphor oil Clove oil	Cinnamonus Cinnamonum camphora Eugenia caryophyllata	Leaves Flower buds	Camphor & cineol Eugenol, eugenyl, acetate, β-caryophyllene, 2-heptanone, ethyl hexanoate, humulenol, α-humulene. calaorene. calamenene	Pragadheesh and others (2013) Mylonas and others (2005), Hossain and others (2014); Chatterjee and Bhattacharjee (2013)
Neem seed oil	Azadirachta indica	Fruits & seeds	Oleic, palmitic, stearic, & linoleic acids	Mandal and Mandal (2011); Devi and Maji (2009); Yang and others (2009b); Abdel-Chaffar and Semmler (2007); Devi and Maii (2011)
Vanilla oil Schinus oil	Vanilla pompona Schinus molle	Green fruit Leaves & berries	V anillin Myrcene, $\alpha$ -phellandrene, $\beta$ - phellandrene, limonene, & p-cymene	Yang and others (2014) Gomes and others (2013); Abdel-Sattar and others (2010); Deveci and others (2010); López and others (2014): Martins and others (2014b)
Gac oil Lavender oil	Momordicac ochinchinensis Lavandula angustifolia	Fruit & seeds Flowers & spikes	Unsaturated fatty acids, <i>β</i> -carotene, & lycopene Linalool, linalyl acetate, lavandulyl acetate, <i>α</i> -teroineol, & oeranyl acetate	Kha and others (2014a), Kha and others (2014b) Vakili and others (2014), Chograni and others (2010); Fakhari and others (2005)
Pimento oil	Pimenta dioica	Leaves & berries	Eugenol, methyl eugenol	Priya and others (2012); Dima and others (2014); Zahka and others (2009)
Pomegranate oil	Punica granatum	Fruit seeds	Punicic, palmitic, stearic, oleic, & linoleic acids	Fazaeli and others (2013); Abbasi and others (2008a);
Zanthoxylum oil	Zanthoxylum limonella	Fruit	Limonene, terpin-4-ol, & sabinene	Itthipanichpong and others (2002); Das and others (2003); Maii and Hussain (2009)
Cumin oil	Cuminum cyminum	Seeds	$eta$ -Pinene, p-cymene, $\gamma$ -terpinene, cuminaldehyde, 1,3-p-mentha, 3-p-menthen-7-al, & cuminyl alcohol	Allahghadring, and others (2010); Sahana and others (2011); Kanakdande and others (2007); Chen and others (2007).
Black pepper seed oil	Piper nigrum	Seeds	eta-caryophyllene, $lpha$ -Pinene, $eta$ -pinene, limonene, sabinene, 3-carene	Jeena and others (2014); Shaikh and others (2006); Bagheri and others (2014)

Evening primrose oil. Evening primrose oil is being used in increasing amounts in nutritional and pharmaceutical preparations. It contains 9% y-linolenic acid, 74% LA, and 7% oleic acid in the lipid profile (Table 1), about 7952 mg  $\beta$ -siosterol and 883 mg campesterol per 1 kg oil in the phytosterol fraction, and about 55 mg phenolic molecules per 1 kg oil (Montserrat-de la Paz and others 2014).  $\gamma$ -Linolenic acid is required by the body cells for maintenance of normal cell structure and regulation of blood lipids and also responsible for many other physiological effects, such as hypocholesterolemic effect, therapeutic effects in atopic eczema, diabetic neuropathy, rheumatoid arthritis, and premenstrual pains (Liu and Yang 2011; Montserrat-de la Paz and others 2014). Recently, it was found that  $\beta$ -sitosterol and campesterol have a cancerprotective role (Montserrat-de la Paz and others 2015). Liu and Yang (2011) reported microencapsulation of evening primrose oil with GA, MD, and/or SC individually or in combination by spraydrying, showed that the microcapsules with single wall material were relatively more susceptible to oxidation than those with multiple wall materials.

Sunflower oil. Sunflower oil is rich in long-chain monounsaturated fatty acid (MUFA) and PUFAs, with the content greater than that of soybean oil, palm oil, mustard oil, and coconut oil. Sunflower oil contains about 46% LA ( $\omega$ -6) and 45% oleic acid ( $\omega$ -9) in the fatty acid profile. In view of potential health benefits of  $\omega$ -6 and  $\omega$ -3 fatty acids, sunflower oil and linseed oil mixtures have been studied at different dietary ratios (Eiben and others 2010). Sunflower oil also has high contents of tocopherols and carotenoids and contains lecithin (Eiben and others 2010). The oil possesses antiinflammatory, anticancer, cardiovascular, and skin-health benefits (Pal 2011) and is effective against bronchial, laryngeal, and pulmonary infections. Spray-drying microencapsulation of sunflower oil increased the oxidative stability (Roccia and others 2014), with high sunflower oil content retained in microcapsules with MD and hydroxypropylmethylcellulose used as wall materials (Roccia and others 2014). Sunflower oil has been microencapsulated by sprav-drying in the matrix using trehalose and whey protein isolate (WPI) or SC. Using trehalose improved the final flowability, which additionally enables increasing the oxidative stability of microencapsulated oil (Domian and others 2014). Sunflower oil was also used to dissolve astaxantin, a type of carotenoid found in marine animals, and encapsulated with blends of milk protein and fiber by spray-drying, in order to enchance the carotenoid stability and application in food systems (Shen and Quek 2014).

**Kenaf seed oil.** Kenaf seed oil is rich in PUFAs (Table 1). ALA in the seed oil acts with anti-inflammatory and antithrombotic activity in the body (Williams and others 2007) and phytosterols have cholesterol lowering ability (Nyam and others 2009). Moreover, it contains considerable amounts of other natural antioxidants (Ng and others 2013a). Microencapsulation was shown to protect kenaf seed oil against oxidation, as well as to prevent the degradation with higher total phenolic contents and to prolong the shelf-life in kenaf seed oil (Ng and others 2013a,b; Razmkhah and others 2013).

**Red pepper seed oil.** Red pepper seeds contain protein, oil, and fiber along with several amino acids. Today, China is the world's largest red pepper producer. Red pepper seeds contain 25% oil and fatty acids, which mainly consist of LA, oleic acid, hexadecanoic acid, stearic acid, and linolenic acid (Table 1). The contents of LA and oleic acid can reach over 90% among the unsaturated fatty acids, which are fundamental to human health and can reduce LDL cholesterol. Some medical investigators consider that red pepper seeds can take precautions against cardiovascular disease and reg-

ulate the central nervous system. Red pepper seeds also contain various minerals and the vitamins A, D, E, and K, and its high level of vitamin E is especially helpful to health care and anti-aging (Ozyildiz and others 2013; Wang and others 2014b). Microencapsulation of ozonated red pepper seed oil for the preparation of functional textile material gave higher antimicrobial activity than nonmicroencapsulated oil (Ozyildiz and others 2013).

**Jojoba oil.** Jojoba oil is a wax that acts directly against aging, moisturizes dry skin, nourishes hair, and prevents the accumulation of sebum. It helps in the renewal process of cells and increases skin elasticity, smoothness, and firmness. It is constituted of eicosanoic acid (66% to 71%), docosanoic acid (14% to 20%), and oleic acid (10% to 13%). Jojoba oil also contains ceramides and flavonoids (Abdel-Mageed and others 2014) and vitamin E, which acts as an antioxidant and protects the skin against free radicals (Jaafar and others 2012). The jojoba oil microcapsules presented controlled release over time when diffusing of jojoba oil, microencapsulated onto compression garment, through a pig skin (Jaafar and others 2012).

Nut oils. A nut is a fruit composed of a hard shell and a seed within. Generally, nuts have a high oil content. Nut oil has a high content of  $\omega$ -6 and  $\omega$ -3 PUFAs (Li and others 2007). Walnuts contains 60% to 65% oil, with LA as the major fatty acid, followed by oleic, linolenic, and palmitic acids (Pereira and others 2008). Phenols, tocopherols, and phytosterols have also been found in walnut oil (Yang and others 2009a). It gives protection against certain types of cancer and also decreases the risk of coronary heart disease (Yang and others 2009a). Calvo and others (2011) reported that the microencapsulation process of walnut oil by SC, carboxymethylcellulose (CMC), and MD did not affecte the PUFA fatty profile but did extend the shelf-life (oil stability for at least 5 mo) regardless of the use of antioxidant additives.

**Olive oil.** Olive oil is of dietetic importance, containing 56% to 87% of MUFA, 8% to 25% of saturated fatty acids, and 8% to 22% of PUFAs (Koutsopoulos and others 2008), also essential vitamins and polyphenols that contribute to the stability of the oil and may have anti-inflammatory and anti-atherosclerotic properties (Lockyer and Rowland 2014). In addition, their potential health benefits include rapid digestibility, antiulcer and anti-aging properties, stress and plasma cholesterol lowering effects, reduced likelihood of developing acute coronary syndrome and cancers, and therapeutic potentials for type 1 and 2 diabetes (Whayne 2010; Perona and Botham 2013). It was found that microencapsulation of olive oil using SC as wall material extended the oil shelf-life without altering the fatty acid profile (Calvo and others 2010, 2012).

Palm oil. Red palm oil is one of the richest sources of vitamin E (tocotrienols 70% and tocopherols 30%; Mukherjee and Mitra 2009). It also contains carotenoids of which 80% to 90% is present as  $\alpha$ -carotene and  $\beta$ -carotene (Benadé 2013). In addition, it contains appreciable amounts of phospholipids, ubiquinones, sterols, and squalene (Choo and others 2005). Oil palm fruits have also been identified as a rich source of phenolic compounds (Sambanthamurthi and others 2011). It is unique in that it contains equivalent amounts of saturated and unsaturated fats (Table 1; Sambanthamurthi and others 2000). Recently, it has been found to have potent protective effects against chronic diseases, that is, cardiovascular disease, diabetes, and cancer (Sambanthamurthi and others 2011; Benadé 2013; Che Idris and others 2014). Dian and others (1996) used red palm olein and stearin in the ratio of 60:40, along with SC as wall material, to prepare microcapsules for improved characteristics.

Sea buckthorn oil. Sea buckthorn seed and pulp oils have been traditionally used for treating skin problems due to its abundance in the content of  $\omega$ -7 unsaturated fatty acid (Ito and others 2014). Buckthorn seed oil is also rich in carotenoids, tocopherols, and phytosterols. Additionally, it contains high levels of the 2 essential fatty acids linoleic (34%) and ALA (25%). These make it one of the most versatile natural oils (Yang and Kallio 2001; Basu and others 2007). Buckthorn seed oil reportedly has a therapeutic role in atopic dermatitis, gastric ulcers, and cardiovascular diseases (Eccleston and others 2002; Xing and others 2002; Basu and others 2007; Ito and others 2014). It was found that encapsulation using GA, MD, and MS improved oxidative stability and prolonged the shelf-life of sea buckthorn seed oil from 1 wk to 2 mo at 20 °C (Partanen and others 2005, 2002).

Avocado oil. Avocado oil has a unique flavor and color. It contains a high concentration (70%) of oleic (MUFA), vitamins, minerals, and phystosterols (Requejo and others 2003; Ozdemir and Topuz 2004). Its phytochemicals are known to provide various positive health effects on serum cholesterol and lipoproteins, coronary heart disease, cancer, and diabetes (Requejo and others 2003). Therefore, avocado oil is widely accepted in food, pharmaceutical, and cosmetic products. Bae and Lee (2008) found that microencapsulation of avocado oil by WPI and MD improved its oxidative stability to some extent.

Wheat germ oil. Wheat germ is a rich source of both tocopherols and tocotrienols. It contains about 8% to 14% oil. Its crude oil is usually high in unsaponifiable matters (2% to 6%). It has been used successfully as an oil phase in emulsion preparations by using sodium alginate (SA) as a wall material (Chan and others 2000; Megahad and El Kinawy 2002).

#### **Essential oils**

Essential oils are volatile, complex mixtures of compounds characterized by a strong odor, and they are formed by aromatic plants as secondary metabolites. Mostly, they possess various biological properties. Essential oils could be considered suitable substitutions to chemical additives for use in the food industry. Compositions of essential oils may be changed as a result of oxidation, chemical interactions, or volatilization. To limit the composition degradation/loss during processing and storage, and to control the delivery of the compound at the desired time and site, encapsulation is beneficial prior to use in foods or beverages (Beirão da Costa and others 2013). Table 1 shows the commonly used oils, their source, extraction, and their major components. The following oils will be listed by source of oil extraction.

Leaves Oregano oil. Oregano essential oils contain more than 30 ingredients, including carvacrol, thymol,  $\alpha$ -terpinene,  $\gamma$ terpinene, terpinen-4-ol, *p*-cymene,  $\alpha$ -terpineol, and sabinene. The first 2 components, constituting about 78% to 82% of the total oil, are phenolic compounds and principally responsible for the characteristic odor and antioxidant and antimicrobial activities (Olmedo and others 2014; Muriel-Galet and others 2015). Supplementation with oregano oil increased the retention of  $\alpha$ tocopherol in meat, which was positively correlated with the supplementation level (Botsoglou and others 2003). Oregano oil is one of the most effective oils for antimicrobial control and its phenolic components have been shown to permeate and depolarize the bacterial cytoplasmic membrane, thus leading to cell death. The antimicrobial efficacy of essential oil is in the order: oregano / clove / coriander / cinnamon > thyme > mint > rosemary > mustard > cilantro / sage (Rodriguez-Garcia and others 2015). Addition of the oil in organic cottage cheese decreased chemical

deterioration during storage (Asensio and others 2015). Moreover, oregano essential oils possess antifungal and insecticidal properties and can also be used for the prevention of neurodegenerative disorders (Almeida and others 2013).

Oregano oil is used as a food ingredient due to its preservative effects and pleasant flavor. However, due to the hydrophobic nature of essential oils, organic compounds from a food matrix may interfere with essential oils to reduce their sterilizing effect and/or shorten the duration of effectiveness (Bhargava and others 2015). Together with instability and volatilization, encapsulation of oregano essential oil has been widely investigated. Tween 80, a food-grade nonionic surfactant, can stabilize oregano essential oil nanoemulsions when prepared by a high-energy ultrasound approach. It reduced Listeria monocytogenes, Salmonella typhimurium, and Escherichia coli O157:H7 on fresh lettuce (Bhargava and others 2015). Oregano essential oil has been encapsulated in skimmed milk powder (SMP) and WPC particles, rice starch porous spheres and inulin (IN), and gelatin/sucrose capsules prepared by spraydrying (Baranauskienė and others 2006; Beirão da Costa and others 2012). When encapsulated in SMP and WPC matrixes, the changes in the composition of flavor compounds during encapsulation were considerably smaller for oregano oil as compared with aroma extracts of sweet marjoram and citronella (Baranauskienė and others 2006). Gelatine/sucrose microparticles exhibited high antimicrobial and antioxidant activity, while IN and rice starch microencapsulates ensured higher stability (Beirão da Costa and others 2012). The oil release behaviors were related to the structure and constituents of the microcapsules and the methodology applied to produce them (Beirão da Costa and others 2012, 2013). By using supercritical fluid technology, oregano oil was impregnated in microspheres of native sorghum and rice starch prolonging the oil antioxidant activity during storage (Almeida and others 2013).

*Mint oil.* Mint oil is a complex mixture of comparatively volatile and labile components. It is a popular flavoring agent in food (Zhong and others 2009), perfumery, and pharmaceutical industries. The most abundant component of mint oil is menthol (Lawrence 1997). It has been used since ancient times for the treatment of many digestive tract problems and in culinary applications. It offers strong antimicrobial (Iscan and others 2002), anticancer (Kumar and others 2004), and antioxidant (İnan and others 2012) activities. Microencapsulation increased the retention of mint oil and its major compounds, mainly menthol and isomenthol (Hwang and others 2006; Sarkar and others 2013, 2012). Dong and others (2011) found that only 7% of peppermint oil was released from microcapsules prepared from gelatin and GA during 40 d of storage in cold water, thereby showing excellent storage stability.

*Basil oil.* Basil oil has emerged as a source of powerful medicinal value (Baliga and others 2013). Its main components of phenolic and terpenoid derivatives include methyl eugenol (42.58%) followed by caryophyllene (26.88%) and eugenol (10.66%). They offers good antioxidant (Chanwitheesuk and others 2005), antibacterial (Burt 2004), antifungal (Kumar and others 2010), antihelminthic (Bihari and others 2010), anti-inflammatory (Singh and others 2007a), antitussive (Nadig and Laxmi 2005), and anticancer properties (Shimizu and others 2013). They can also inhibit cholesterol synthesis (Khanna and others 2010) and improve digestive performance (Baliga and others 2013). Sutaphanit and Chitprasert (2014) found that microencapsulation of basil oil using gelatin provided protection against physical and chemical loss under accelerated storage conditions at 60 °C for 49 d. However,

a small decrease was observed in its retention rate and antioxidant activity.

Rosemary oil. Rosmarinus officinalis L. is the most used aromatic and medicinal plant worldwide, because of its essential oil and phenolic compounds (Rozman and Jersek 2009). The latest research related to rosemary essential oil has mainly focused on its antibacterial (Jiang and others 2011), antifungal (Soylu and others 2010), insecticidal (Zoubiri and Baaliouamer 2011), anticancer (Degner and others 2009), astringent, antiseptic, gastric, carminative, antiviral, anti-inflammatory, and antioxidant properties (Barni and others 2012). Moreover, it also helps in cognition improving (Moss and others 2003). Microencapsulation of rosemary oil improved functional activity with high retention volatiles (1,8-cineol, camphor, and  $\alpha$ -pinene; de Barros Fernandes and others 2014; Fernandes and others 2014). The chemical composition of encapsulated rosemary oil remains unchanged (Fernandes and others 2013).

Lippia sidoides oil. L. sidoides, an aromatic herb, is popularly known as "alecrim pimenta." This species is used effectively to treat skin, scalp infections, mouth, throat, and gastroenteric mainly due to its antiseptic, anti-infective, and antimicrobial properties (Costa and others 2001; Pascual and others 2001). Its essential oil has antibacterial and antifungal (Botelho and others 2007), insecticidal (de Lima and others 2013), antihelminthic (Camurca-Vasconcelos and others 2007), and acaricidal properties (Cavalcanti and others 2010). Moreover, it is also used in the commercial production of perfumes, creams, lotions, and deodorants (Botelho and others 2007). Fernandes and others (2008) demonstrated that microencapsulation of L. sidoides essential oil using MD and GA produced a powdered product containing a high concentration of thymol, a major constituent of the essential oil, compared to unencapsulated oil, and it showed important antifungal activity.

Lemongrass oil. Cymbopogon citratus (DC) Stapf (Poaceae), commonly known as lemongrass, is widely used in traditional and folk medicine (Kpoviessi and others 2014). Lemongrass essential oil has antimicrobial activity against a diverse range of microorganisms, including, yeasts, and Gram-positive and Gram-negative bacteria (Naik and others 2010). Indeed, lemongrass oil is effective against generalized anxiety disorder and epilepsy (Costa and others 2011). In addition to its broad spectrum of fungitoxicity, considerable interest has developed in the application of lemongrass oil for the preservation of stored food crops (Mishra and Dubey 1994). Leimann and others (2009) found that microencapsulation of lemongrass oil by poly(vinyl alcohol) (PVA), protected oil from deterioration and maintained its antimicrobial activity.

Lemon myrtle oil. Australian essential oils of Backhousia citriodora (lemon myrtle) contains typically 95% citral. It possesses a sweet aroma, intensive lemon-flavor that could be used as a lemon-flavor replacement in milk-based foods (Southwell and others 2000; Forbes-Smith and Paton 2002). It has an excellent antimicrobial activity (Burke and others 2004) and may also offer antioxidant properties (Konczak and others 2010). Due to favorable characteristics, it has been widely used in foods, cosmetics, and medicines. Huynh and others (2008) successfully used WPC and MD to encapsulate lemon myrtle oil.

*Cinnamon leaf oil.* Cinnamon leaf oil is recognized for its flavor and aroma in addition to it antimicrobial properties (Singh and others 2007b; Ayala-Zavala and others 2008). Antifungal and antioxidant properties of cinnamon leaf oil are due to volatile components such as cinnamaldehyde and eugenol (Combrinck and others 2011). It has been found to have antimicrobial (Matan and others 2006), antidiabetic (Ping and others 2010), and anti-inflammatory

properties (Gunawardena and others 2014). The U.S. Food and Drug Administration regards cinnamon oil as a Generally Recognized as Safe compound (Tzortzakis 2009). Microencapsulation of cinnamon leaf and garlic oil by  $\beta$ -cyclodextrin displayed good antifungal activity against *Alternaria alternata*. Cinnamon leaf and garlic oil microcapsules could have important applications in the food industry due to improved stability, solubility, and bioavailability (Ayala-Zavala and others 2008).

*Tea tree oil.* Australian endemic plants produce a wide range of essential oils such as tea tree (*Melaleuca alternifolia*) oil, which has been employed for its germicidal and antibacterial activity (Thomsen and others 2013). Moreover, it is used in consumer health products including topical antiseptics, mouthwashes, acne treatments, oral *candidiasis*, herpes *labialis*, and dandruff. Microencapsulation of tea tree oil using melamine maintained antimicrobial activity and was used as a biocide for footwear applications (Sanchez-Navarro and others 2011).

Thyme oil. Thyme is a phytogenic feed additive and a known source of essential oils. Thyme essential oil is widely used in the flavor and food industries and also in the manufacturing of perfumes and cosmetics. Its antioxidant and antimicrobial activities are mainly attributed to the presence of carvacrol, cinnamaldehyde, thymol, geraniol, and eugenol (Sipailiene and others 2006; Navarrete and others 2010). As a pharmaceutical compound, thymol and carvacrol are used in mouthwashes, soaps, and creams (Sipailiene and others 2006). Recently, it has been found that incorporating thyme essential oil into edible films improved the shelf-life and safety of ready-to-eat foods (Jouki and others 2014b). The melamine—formaldehyde microcapsules had good thermal resistance, a smooth surface, and high insect-repellent efficacy (Chung and others 2013).

*Camphor oil*. Camphor oil is valued by many peoples, particularly the Chinese, and used for medicinal purposes. Therapeutic properties of camphor oil are antifungal, analgesic, antidepressant, anti-inflammatory, antiseptic, carminative, diuretic, febrifuge, hypertensive, insecticidal, laxative, rubefacient, stimulant, sudorific, vermifuge, and vulnerary (Deng and others 2005; Pragadheesh and others 2013). Recently, a new and safe product was developed from camphor oil for controlling poultry lice (Khater and others 2014). Chang and others (2006) encapsulated camphor oil using gelatin-GA microcapsules prepared by complex coacervation to achieve encapsulation efficiency of 99.6 wt% at an optimal oil/wall volume ratio of 0.75 and found that the sustained oil release amount and the release rate depended on the quantity of added polystyrene.

Schinus molle oil. S. molle (L.) (Anacardiaceae) is a highly aromatic evergreen tree, known as a source of essential oils with a pleasant spicy scent (Murray and others 2005; Gomes and others 2013). Its essential oils have antibacterial, antifungal, antiinflammatory, cytotoxic, insecticidal (Abdel-Sattar and others 2010; Deveci and others 2010; López and others 2014), antioxidant, and antimicrobial properties (Martins and others 2014b). Microencapsulation of the S. molle Rev L. (Anacardiaceae) leaves essential oil, using MD and GA, was developed to control the release of active ingredients and protecting them from the external environment during product application and storage. It showed that the insecticidal effect was quite persistent, with the synchronous improvement of its insecticidal potential on *Haematobia irritans* (López and others 2014).

*Pimento oil.* Pimento essential oil is extracted from leaves and berries of the *Pimenta dioica* tree. It is used in foods, especially in meat, cosmetics, and pharmaceutics to treat diabetes, high

blood pressure, neuralgia, and stress (Priya and others 2012). The pimento essential oil extracted from berries of *P. dioica* (L) Merr. contained 23 components including eugenol (68.06%) and methyl eugenol (9.37%; Dima and others 2014). These compounds have significant antifungal activity, especially against dangerous pathogenic and toxinogenic fungi (Zabka and others 2009). Microencapsulation of pimento essential oil using chitosan and  $\kappa$ -carrageenan exhibited antimicrobial activity against *Candida utilis*, *Bacillus cereus*, and *Bacillus subtilis*, suggesting that *P. dioica* essential oil encapsulated in chitosan and chitosan/ $\kappa$ -carrageenan microspheres can be used in the meat industry to increase the functionality of meat products (Dima and others 2014).

**Seeds** *Cardamom oil.* Cardamom oil has potential applications as an antimicrobial, antibacterial, antioxidant (Sengottuvelu 2011), anticancer, antiseptic, antispasmodic, carminative, cephalic, digestive, diuretic, expectorant, stimulant, and stomachic (Kumari and Dutta 2014). Moreover, it is an efficient skin permeation enhancer for certain drugs agents (Sengottuvelu 2011). The major compounds present in the cardamom oleoresin are 1,8-cineole and  $\alpha$ -terpinyl acetate comprising about 66% of the total volatiles. Microencapsulation of cardamom oil using blends of GA, MD, and MS increased the stability of volatiles including 1,8-cineole and  $\alpha$ -terpinyl acetate (Beristain and others 2001; Krishnan and others 2005).

Garden cress oil. Garden cress is an edible herb and its seeds contain about 24% oil, of which 32% to 34% is ALA (Moser and others 2009; Umesha and others 2015). Garden cress oil has very high amounts of tocopherols (1699 mg/kg), which exhibit antioxidant potency (Moser and others 2009). Encapsulation did not affect fatty-acid composition of garden cress oil with respect to ALA levels in microcapsules, but it did confer good oxidative stability and protection to the oil (Umesha and others 2013, 2015).

Coffee oil. Green coffee oil is usually used in the cosmetic industry since its fatty acid composition has important emollient properties. The oil has about 80% triglycerides and high amounts of unsaponifiable compounds (Silva and others 2014). This unsaponifiable fraction contains the diterpenes kahweol and cafestol in free form and esterified with fatty acids, predominantly with LA (46.3% and 44%), palmitic acid (30.2% and 31.3%), oleic acid (10.6% and 12.5%), and stearic acid (8% and 5.9%), extracted from the species Coffea canephora and Coffea arabica, respectively. The oil from C. arabica is more suitable for applications in cosmetics, thus offering the highest sun protection factor (SPF; Wagemaker and others 2011), especially for avoiding UVB radiation (280-320 nm) effects on skin, and also as an anticarcinogenic, anti-inflammatory, and antioxidant agent (Silva and others 2014). Microencapsulation of green coffee oil exhibited higher oxidative stability than unencapsulated oil and maintained the SPF with high amounts of di-terpenes. Morever, encapsulation of this oil improves its application in powdered cosmetics and reduces allergenic effects of cinnamic acid when applied directly to the skin (Frascareli and others 2012; Carvalho and others 2013; Silva and others 2014).

*Pitaya seed oil. Hylocereus polyrhizus*, commonly known as red pitaya or dragon fruit, is a variety of cactus fruit that has red-skinned fruit with red-violet (reddish purple to purplish red) color in the fruit flesh (Lim and others 2010; Hor and others 2012). Pitaya seed oil can serve as a potential source of natural antioxidants such as phenols, sterols, and tocopherols (Tenore and others 2012). Pitaya seed oil may assist in the prevention of chronic diseases (Wybraniec and Mizrahi 2002; Wu and others 2006). Moreover, it has been reported to exhibit antimicrobial effects (Du and others 2011). Lim and others (2012) found that encapsulation of red-

fleshed pitaya seed oil using SC and whey protein increased its oxidation stability.

*Caraway oil.* Caraway seeds are used as a spice in food due to its pleasant flavor. Caraway essential oil has antioxidant, insecticidal, antibacterial, fungicidal, acaricidal, molluscicidal, and larvicidal activities (Laribi and others 2009). Caraway essential oil is important in pharmaceutical applications and also in human medicine due to its diuretic (Lahlou and others 2007), antihyperglycemic (Tahraoui and others 2007), antihyperchlolesterol (Lemhadri and others 2006), and anticancerous properties. Recently, it was found that caraway oil poultices were efficient in the treatment of irritable bowel syndrome (Langhorst and others 2014). Microencapsulation of caraway (*Carum carvi* L.) oil with WPC and surface-active carbohydrates increased the retention of volatile compounds during spray-drying beside enhancing the protective properties of solidified capsules against oxidation and release of volatiles during storage (Bylaite and others 2001).

Neem seed oil. Neem seed oil is a commercialized product derived from fruits of the neem tree, also named margoaa oil. Neem oil has been used in the treatment of various inflammation-related diseases, malaria, skin disorders, ulcers, and promotion of wound healing due to its antibacterial, antifungal, and antiparasitic activities (Abdel-Ghaffar and Semmler 2007; Yang and others 2009b; Mandal and Mandal 2011). Devi and Maji (2009) successfully used chitosan-carrageenan polyelectrolyte complex as an efficient matrix to encapsulate neem seed oil. The structures of the microcapsules were found to change from free-flowing to bursting as the loading of neem seed oil increased (Devi and Maji 2011).

Cumin oil. Cumin (Cuminum cyminum L.; Umbelliferae family) is an annual plant and is one of the most commonly used spices and condiments in food preparations (Allahghadri and others 2010). Cumin provides additional taste and flavor to foods and is also used in traditional medicine (Hashemi and others 2008; Sahana and others 2011). Cumin volatile oil contains over a dozen chemical components including terpenes (such as  $\beta$ -pinene, p-cymene, and  $\gamma$ -terpinene), aldehydes (cuminaldehyde, 1,3-pmentha, and 3-p-menthen-7-al), and terpene alcohol (cuminyl alcohol; Kanakdande and others 2007). In recent years, other functional activities of cumin oils have been extensively studied, including antimicrobial, anticancer, antidiabetic, and antioxidant effects (Allahghadri and others 2010; Sahana and others 2011; Chen and others 2014). Kanakdande and others (2007) found that microencapsulation of cumin oleoresin by GA/MD/MS (4/6:1/6:1/6) blends was better than GA itself in stabilizing the volatile contents (cuminaldehyde,  $\gamma$ -terpinene, and p-cymene), which warrants that such microcapsules can be used in the food industry.

Black pepper seed oil. Black pepper (*Piper nigrum*) seed is the most important and most widely used spice in the world and is known as the "King of Spices" (Ravindran and Kallupurackal 2012). Black pepper oils have antioxidant, radical-scavenging (Bagheri and others 2014; Jeena and others 2014), anti-inflammatory, and antinociceptive properties (Jeena and others 2014). It has been found that encapsulation of black pepper oleoresin using GA was better than MS in protecting piperine (Shaikh and others 2006).

**Fruits** *Citrus oil.* Citrus fruits are widely cultivated and consumed throughout the world. Their essential oils are a mixture of volatile compounds consisting mainly of monoterpene hydrocarbons and exist especially in citrus peels, flowers, and leaves. The citrus peel, which represents roughly half of the fruit mass, is a rich source of bioactive compounds (Li and others 2006). The citrus outer peel, known as flavedo, has a large number of very small glands, each containing a minute drop of essential oils

(Frerot and Decorzant 2004). Mandarin peel oil is composed of 3 major compounds, (limonene, limonene/ $\gamma$ -terpinene, and linalyl acetate/limonene, Table 1), while leaf (petitgrain) oil consists of 3 other compounds (sabinene/linalool,  $\gamma$ -terpinene/linalool, and methyl N-methylanthranilate; Lota and others 2001). Citrus essential oils have been used in various industries including cosmetic, pharmaceutical, food product, and tobacco industries because of their special flavor and fragrance as well as biological activities. The citrus oils present the biggest potential contribution to the content of furocoumarin in fragrance products (Frerot and Decorzant 2004). Citrus essential oils show antioxidant activity, due to the presence of volatile components, in which geraniol, terpinolene, and  $\gamma$ -terpinene have a 1,1-diphenyl-2-picrylhydrazyl randicalscavenging effect 3.5 times as strong as that (Choi and others 2000). The major components in kaffir lime oil are  $\beta$ -pinene (30.6%) and limonene (29.2%) and have antibacterial properties (Adamiec and others 2012). Moreover, lemon, mandarin, grapefruit, and orange essential oils have antifungal activities (Viuda-Martos and others 2008). Orange oil also has strong fumigant and contact activities against Sitophilus zeamais and Tribolium castaneum adults, showing stronger activity in the contact assay than basil oil (Kim and Lee 2014). In citrus essential oils, volatile compounds are not stable and can be oxidized and deteriorated when exposed to high temperature, oxygen, and humidity. Encapsulation of the oils has been widely studied using spray-drying technique. When encapsulating mandarin peel oil, using GA and MD mixtures, 96.0% of volatile oil retention and 99.4% of microencapsulation efficiency could be obtained at an inlet air temperature of 200 °C and an exit air temperature of 80 °C (Bringas-Lantigua and others 2011). In the case of kaffir lime oil, the combination of konjac glucomannan (KGM) and GA as wall material could enhance the yield and retention of total oil in the microcapsules more than using KGM alone (Adamiec and others 2012). Beristain and others (2002) found that GA or its mixtures with mesquite gum at a 3:2 weight ratio could encapsulate 93.5% orange peel oil, while MD and mesquite gum mixtures retained 84.6%, they are better than mesquite gum did (80.5%; Beristain and Vernoncarter 1994). Mesquite gum-encapsulated orange peel oils showed very good stability against oxidation when stored in water activity 0.628 at 35 °C for 30 d (Beristain and others 2002).

Pterodon emarginatus oil. The essential oil extracted from the fruits of the genus Pterodon (P) has been widely studied. It exhibits numerous pharmacological activities proven to treat bronchitis and tonsillitis (Evangelista and others 2007), anti-Chagas' disease caused by Trypanosoma cruzi (Menna-Barreto and others 2008), and shows antimicrobial (Dutra and others 2009), anti-inflammatory, antinociceptive (Barbosa Raposo and others 2011), and antioxidative properties (Barbosa Raposo and others 2011). It was observed that  $\beta$ -caryophyllene (sesquiterpene hydrocarbon) was the major compound (Table 1) and possible marker for the volatile fraction of this species. Microencapsulation of essential oil from the fruits of *P. emarginatus* using GA and MD exhibited high stability during storage with, 70.55% retention of  $\beta$ -caryophyllene over 45 d. In addition, microcapsules exhibited favorable functional properties and possessed good potential for use in herbal medicine because of their ability to conserve and protect the essential oil from degradation and evaporation (Alves and others 2014).

*Vanilla oil*. Vanilla oil is extracted from the vanilla plant, specifically from its green fruit. It is widely used in perfume, body oil, and other holistic health or beauty treatments (Yang and others 2014). The major compound of vanilla oil is vanillin and used as an antioxidant, anticarcinogenic, and antimutagenic agent due

to its phenolic character (Sinha and others 2008). Microencapsulation of vanilla oil using chitosan and GA through complex coacervation enhanced its retention and thermostability to serve as a high-quality food additive (Yang and others 2014).

Gat oil. Gac aril oil contains unusually high levels of carotenoids, especially  $\beta$ -carotene and lycopene (Kha and others 2014a). They help to prevent xerophthalmia, hemeralopia, photopsia, and weakness of eyesight. Moreover, it increases blood red cells of the body and lowers prostate cancer risk. Also, it can be used as a supplement (Vuong and others 2002). In addition, significant amounts of unsaturated fatty acids are found in the arils (Vuong and others 2002). It has been found that gac oil microcapsules could be incorporated successfully into various foods for consumers to benefit from the food nutrients and enjoy the attractive red-yellow color (Kha and others 2014b).

Pomegranate oil. Pomegranate (Punica granatum L.) is one of the oldest known edible fruits that contain the highest concentration of total polyphenols in comparison with other fruits studied (Fazaeli and others 2013). Pomegranate seed oil consists of 65% to 80% conjugated fatty acids (Abbasi and others 2008a) and has the highest botanical concentration of a sex hormone (estrone) at 17 mg/kg dry seed (Abbasi and others 2008b). It was reported to present biological properties (Eikani and others 2012), such as antioxidant and eicosanoid enzyme inhibition properties (Qu and others 2010), immune function and lipid metabolism (Yamasaki and others 2006), estrogen content (Tong and others 2006), skin photoaging inhibition effect (Park and others 2010), lipoperoxidation and activity of antioxidant enzymes (de Melo and others 2010), toxicological evaluation (Meerts and others 2009), and protective effect against gentamicin-induced nephrotoxicity (Boroushaki and others 2014). Goula and Adamopoulos (2012) successfully spray-dried pomegranate oil with 95.6% encapsulation efficiency using SMP as an encapsulating agent.

Zanthoxylum limonella oil. Z. limonella Alston (Rutaceae) is locally called "Ma-khan." Its roots, stem-barks, stems, and fruits are used for treating stomachache and toothache (Itthipanichpong and others 2002). The essential oil from fruits confers stimulation effect on different smooth muscles (Itthipanichpong and others 2002). Additionally, leaves possess mosquito repellent (Das and others 2003). Z. limonella oil can be encapsulated in chitosangelatin complex microcapsules cross-linked with genipin; and the release rate of oil decreased with the decrease in the oil loading and with the increase of the percentage of genipin and the concentration of chitosan in the chitosan–gelatin mixture (Maji and Hussain 2009).

Flowers Clove oil. Clove oil is obtained by distillation of the flowers, stems, and leaves of the clove tree (Eugenia aromatica or Eugenia caryophyllata; Mylonas and others 2005). Clove essential oil has antimicrobial (Hossain and others 2014; Song and others 2014) and antioxidant activities (Gülçin and others 2012). Humans have used clove oil for centuries, as an anesthetic for toothaches, headaches, and joint pain, and also as a topical analgesic in dentistry (Curtis 1990; Soto and Burhanuddin 1995). Furthermore, it has been used as an aromatherapy oil, mouth sterilizer, or painkiller (Rabenhorst 1996). Clove oil is used worldwide as a food-flavoring agent. It has been reported that clove oil has antilisteric activity in meat and cheese (Menon and Garg 2001). Chatterjee and Bhattacharjee (2013) revealed that microencapsulation of eugenol-rich clove extracts in soybean oil using MD and GA allowed controlled release of the clove extract antioxidants. It is suggested that clove oil could be used as a natural antioxidant in soybean oil.

Encapsulation of marine, vegetable, essential oils ...



Figure 4–Illustration of the 4 emulsion systems (W/O, O/W, W/O/W, and O/W/O).

therapeutic purposes in herbal medicine (Vakili and others 2014). Lavender oil (Lavandula angustifolia) is an essential oil that has been widely used in food, aromatherapy, fragrant balms, salves, perfumes, colognes, soaps, skin lotion, and other cosmetics (Chograni and others 2010), because of its delightful aroma (Pala-Paul and others 2004) and more than 30 components (Fakhari and others 2005). Recently, it has been demonstrated that lavender oil had neuroprotective activity and improved neurologic functions, reduced brain edema, and might have a potential therapeutic effect in stroke patients (Vakili and others 2014). Xiao and others (2014) successfully employed complex coacervation using gelatin and GA as encapsulants to protect lavender oil.

Roots Vetiver oil. Essential oil extracted from roots of vetiver grass, Chrysopogon zizanioides (L.), has been used in perfumery and medicine for a long time due to its aromatic and biological properties. Vetiver oil consists of a complex mixture of sesquiterpene alcohols and hydrocarbons with over 300 compounds. It is used for blending in oriental types of perfumes, cosmetics, and in aromatherapy. Recently, the discovery of new biological activities of vetiver oil and its components, such as antifungal, antibacterial, anticancer, anti-inflammatory, and antioxidant activities, make vetiver extracts promising candidates for application in the pharmaceutical industry (Danh and others 2009). Prata and others (2008) studied release properties of chemically and enzymatically cross-linked gelatin-GA microparticles prepared through complex coacervation. Vetiver essential oil containing a fluorescent probe revealed that the cross-linking with glutaraldehyde was more efficient in the retention of the fluorescent compound.

# Microencapsulation Techniques of Oils

Numerous techniques have been used for the microencapsulation of marine, vegetable, and essential oils and are discussed briefly in this review. These techniques include emulsification, spray-drying, coaxial electrospray system, freeze-drying, coacervation, in situ polymerization, extrusion-coating, supercritical fluid technology, and fluidized-bed-coating.

## Emulsification

Emulsions are used in a wide variety of food and pharmaceutical products. Emulsification technology is a key step in the microencapsulation of oils. It is generally applied for the encapsulation of

Lavender oil. Lavender has a variety of cosmetic uses as well as bioactives in aqueous solutions, which can either be used directly in the liquid state or can be dried (spray- or freeze-drying) to form powders after emulsification. Therefore, it is a part of the microencapsulation process. For example, in spray- and freezedrying microencapsulation, the core and wall materials could be prepared by emulsion techniques before the final drying. Emulsion droplets can also be incorporated into the matrix during the extrusion process, or act as templates for coacervation processing. Basically, an emulsion consists of at least 2 immiscible liquids, usually oil and water, with one of the liquids being dispersed as small spherical droplets in the other. A system that consists of oil droplets dispersed in an aqueous phase is called an oil-in-water (O/W) emulsion, whereas a system that consists of water droplets dispersed in an oil phase is called a water-in-oil (W/O) emulsion. Multiple emulsions, such as oil-in-water-in-oil (O/W/O) or water-in-oil-in-water (W/O/W) emulsions have also been developed (Figure 4). To obtain a kinetically stable solution, emulsifiers or texture modifiers are commonly added in the emulsion system. The diameters of the emulsion droplets in food systems range from 0.1 to 100  $\mu$ m (Fang and Bhandari 2010). The use of this technology for delivering food components and nutraceuticals has been comprehensively reviewed by McClements and others (2009). The emulsions are prepared by homogenizing oil, water, and emulsifier together using a mechanical device known as a homogenizer (high shear mixer, high-pressure homogenizer, colloid mill, sonicator, or membrane homogenizer). The O/W emulsion consists of small oil droplets dispersed in an aqueous medium, with the oil droplets being surrounded by a thin interfacial layer consisting of emulsifier molecules. The advantages of these systems are relative ease of preparation and low cost, but have the drawbacks of physical instability (when exposed to heating, chilling, freezing, drying, pH extremes, and high mineral concentrations) and limited control release (McClements and others 2009). Chan and others (2000) used emulsification technology successfully in microencapsulation of wheat germ oil and evening primrose oil using SA with a maximum microencapsulation efficiency of about 88% for pharmaceutical applications. Additional functionalities of the emulsions can be achieved by modification of the emulsifiers, such as the use of Maillard reaction products for encapsulation of fish oil. The fish oil was emulsified with heated aqueous mixtures consisting of a carbohydrate source (oligosaccharide, dried glucose syrup, and glucose) and protein (WPI, SPI, SC, or SMP)

and spray-dried for the production of oil powders. The Maillard reaction products increased the encapsulation efficiency and were effective for protecting microencapsulated fish oil and other oils from oxidation (Augustin and others 2006).

# Spray-drying

Spray-drying is a low-cost microencapsulation technology commonly used on an industrial scale, which has the attractive advantages of producing microcapsules in a relatively simple, continuous operation and inexpensive, compared to other microencapsulation techniques. Spray-drying is the most commonly used technique for the encapsulation of oils (Table 2). This technique has been successfully used for several decades to encapsulate various oils in the food industry. Recently, several studies have been undertaken to encapsulate the oils by spray-drying in the food (Bao and others 2011; Polavarapu and others 2011; Wan and others 2011a; Wang and others 2011; Aghbashlo and others 2012a,b; Huang and others 2014; Rubilar and others 2012a; Aghbashlo and others 2013a,b; Carneiro and others 2013; Gallardo and others 2013; Karaca and others 2013b; Ng and others 2013b; Sanguansri and others 2013; Tontul and Topuz 2013; Umesha and others 2013; Botrel and others 2014a,b; de Barros Fernandes and others 2014; Domian and others 2014; Fernandes and others 2014; Kha and others 2014b; Liu and others 2014; Shen and Quek 2014; Tatar and others 2014), cosmetics (Huynh and others 2008; Adamiec and others 2012; Frascareli and others 2012; Sarkar and others 2012; Carvalho and others 2013), pharmaceutical (Liu and Yang 2011), and pesticide industries (López and others 2014).

Spray-drying equipment is readily available and production costs are lower than most other methods. Compared to freeze-drying, the cost of spray-drying is 30 to 50 times lower. Spray-drying has been considered as a solution for conventional drying problems because the process has usually proved to not only be efficient, but also economic. However, some energy in terms of heat is wasted during the spray-drying process (Gharsallaoui and others 2007). On the other hand, spray-drying is rapid and reproducible, allowing easy scale-up compared to other encapsulation techniques, justifying its preferred use in the industrial sector (Pu and others 2011; Schafroth and others 2012). The process is flexible, thereby offering substantial variation in the encapsulant matrix and produces particles of good quality.

Spray-drying involves the atomization of emulsions into a drying chamber at a relatively high temperature, which leads to very fast water evaporation and, therefore, crust formed at fast rate and quasi-instantaneous entrapment of oils (Tonon and others 2011). Water removal by spray-drying is the most widely used practice in the food industry to ensure the microbiological stability of products. Furthermore, it helps to obtain a product with specific functional properties, avoid many of the risk of chemical and/or biological degradation, and, finally, reduce the total storage and transport costs (Gharsallaoui and others 2007; Turchiuli and others 2014). The microencapsulation by spray-drying involves 4 stages, as shown in Figure 5. It consists of (i) preparation of the dispersion, (ii) homogenization of the dispersion, (iii) atomization of the emulsion, and (iv) dehydration of the atomized particles. Ordinarily, in the 1st stage the wall materials are dissolving in distilled water with stirring. The solutions are kept overnight at room or refrigerator temperature to ensure full saturation of the polymer molecules and to avoid any changes due to temperature. The core material is added to the solutions with or without the addition of an emulsifier, depending on the emulsifying properties of the wall materials, and all this before entering into the 2nd stage. In the

spray-drying process, the initial emulsion droplets are in the range of 0.1 to 100  $\mu$ m in diameter. For example, spray-drying of kenaf, cardamom, flaxseed, and orange peel oil produced microcapsules with a diameter of 0.13, 2.59, 22.4, and approximately  $2.5\mu m$ , respectively (Beristain and others 2001, 2002; Karaca and others 2013b; Ng and others 2013a). The formed emulsion must be stable over a certain period of time, before the spray-drying step (Liu and others 2001), viscosity should be low enough to prevent air inclusion in the particle, and oil droplets should be rather small (Drusch 2007). Emulsion viscosity and particle size distribution have significant effects on microencapsulation by spray-drying. It is possible formation of elongated and large droplets that adversely affect the drying rate, because of high viscosities, which interfere with the atomization process. (Rosenberg and others 1990). Spray-drying also requires well-adjusted operating conditions, as well as the correct composition of the solution that contains the active principles (Gallo and others 2011; Soliman and others 2013).

In order to obtain high encapsulation efficiency, optimal spraydrying conditions must be used. The feed temperature, air inlet temperature, and air outlet temperature are the main factors in spray-drying that must be optimized (Liu and others 2004). In fact, feed temperature modifies the viscosity and fluidity of the emulsion, its capacity to be homogeneously sprayed is thus negatively affected. When the feed temperature is increased, viscosity and droplets size should be decreased. However, volatility or deterioration of some heat-sensitive components can occur due to high temperatures. To ensure that each sprayed droplet reaches the desired drying level, the feed rate to atomizer is adjusted, before spraying into the drying chamber. Moreover, appropriate adjustments of the air inlet temperature and flow rate is important (Zbicinski and others 2002).

Microencapsulation efficiency can be increased by increasing the concentration of solids (wall materials) in the solution, which can be related to the formation of a shell around the core material (Young and others 1993). Rubilar and others (2012a) reported that microencapsulation efficiency of flaxseed oil increased from 54.6% to 90.7% when used a high concentration of 30% wall material (MD/GA wall type) instead of using 25% wall material (Table 2). In another study, foamed and nonfoamed emulsions of sunflower oil were spray-dried. Results showed that the foaming process reduced the powder density by 50% compared to the powder produced from a nonfoamed emulsion besides increasing the microencapsulation efficiency (Lewandowski and others 2012). Foaming of the emulsion enabled the control of apparent density of the powders.

Depending on the core material and the characteristics desired in the final product, wall materials can be selected from a wide variety of natural (for example, starch and cellulose) and synthetic (for example, nylon, polyethylene, and polyester) polymers. The wall material must be soluble in water at an acceptable level, because almost all spray-drying processes in the food industry are carried out from an aqueous feed formulation (Gouin 2004). It is necessary to choose the wall materials with high efficiency for microencapsulation by spray-drying because they are playing an important role in encapsulation efficiency and microcapsule stability. The criteria for selecting a wall material are mainly based on its physicochemical properties such as solubility, molecular weight, glass/melting transition, crystallinity, diffusibility, and film forming and emulsifying properties. Moreover, the cost should also be considered. Thus, judicious choice of encapsulating material according to the desired application is an important task (Gharsallaoui and others 2007).

					Temperat	ture/pH		The nexticle			
m.         Name offers         Intervision         Intervision         Optimization         Page offersion         Page offersion         Reference           ns         6 - 0 - 0 - 0 - 0 - 0 - 0 - 0 - 0 - 0 -	-	-						size of		: - 1	
m.         6.4         Complexity         Complexity         Control of the complexity         S0 the data (105 c) (135 c	Vall mater concentra	'ial, ation	Ratio of core & wall	Technique	Inlet (°C)	Outlet (°C)	Oxidative stabil- ity∕retention	capsules∕DS (μm)	Applications	Encapsulation efficiency (%)	References
Bit         Idade         Spray dyring         Id ± ± 1         ID ± 1         ID ± 105	atin:sodi lexameta lhate, 5.1	um iphos-	6:04	Complex coacervation	50 then dried at 10	05⁄pH (3.8:7)	OSI (40.16 h)	50 to 80	Food	88.03 to 99.82	Nang and others (2014a)
	, GA-HC	, & HC	1:04	Spray drying	164 ± 2	102 ± 1	HC was more oxidatively stable	1.05 to 4.09	Food	80.9 to 92.3	Fatar and others (2014)
$ \begin{array}{c c c c c c c c c c c c c c c c c c c $	1, WPI/N 1:1), & V 1:1)	AD VPI/IN	1:03	Spray drying	180	WN	WN	18.6, 17.3, & 15.6 for WPI, WPI/MD, & WPI/IN, respectively	Food	WN	3otrel and others (2014a)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	40%		Oil load of 6%	Spray drying	185	NM	NN	9.6 to 15.6	Food	WZ	3otrel and others (2014h)
25% to 05%         101         Complex conservation         50 $\mu$ H d         Model         684 to 1359         Food         16.71 to 92.09         Tamijal and others(2013)           05% 05%         050/1150mL of reace/ying mith 150mL         5nay drying strate         190 $\pm 3$ 90 $\pm 3$ 90 $\pm 3$ 90 $\pm 3$ 20-0ct         Food         5nay drying mith 2013)         Tamijal and others(2013)           WPL mith 150mL         Frees-drying of relation         190 $\pm 3$ 90 $\pm 3$ 90 $\pm 3$ PV (2.98 mEq./Kg)         20-0ct         Food         5pray drying miths (2013)         Tamijal and others(2013)           WPL mith Privith         1:02         Spray drying         140,160, & 180         NM         NM         1.37 to 4.59         Food         59.05 to 85.67         Aphbashlo and others(2013)           Privith Privith         1:02         Spray drying         140,160, & 180         NM         NM         1.37 to 4.59         Food         59.05 to 85.67         Aphbashlo and others           Privith Privith         1:02         Spray drying         140,160, & 100         NM         NM         1.37 to 4.59         Food         79.14         Aphbashlo and others           Privith Privith         1:02         Spray drying         140,180         NM         NM	glucose jlucose natrix,	::dried syrup 1:1:1	MM	Spray drying	180	80	84% to 86% remaining EPA & 85% to 87% remaining DHA/18 mo at	MM	Food	79 to 99	sanguansri and others (2013)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	atin, 1. 5% & A .25% t	25% to G, 0.5%	1:01	Complex coacervation	50/pH 4		WN	6.84 to 13.59	Food	16.71 to 92.09	Famjidi and others (2013)
WPI, I :02         Trecze drying Spray drying         NM 140, 160, & 180         NM NM         NM         1.37 to 4.59         Food         59.05 to 85.67         Aghbashlo and othes           PL, & Plwith         1:02         Spray drying         140, 160, & 180         NM         NM         1.58 to 8.73         Food         59.05 to 85.67         Aghbashlo and othes           0         1:02         Spray drying         140, 160, & 180         NM         NM         1.58 to 8.73         Food         59.05 to 85.67         Aghbashlo and othes           0         1:02         Spray drying         177.23         NM         NM         79.14         Aghbashlo and othes           0         1:02         Spray drying         177.23         NM         NM         79.14         Aghbashlo and othes           Plwith Privith after of the coupling RSM         140:180         77.85:103.20         NM         1.37 to 4.59         Food         79.14         Aghbashlo and othes           Plwith after of the adgrame         59.05 drying         140:180         77.85:103.20         NM         Plwith         79.14         Aghbashlo and othes           Foud         1:02         Spray drying         140:180         77.85:103.20         NM         Plwith         2012.01         2012.01 <td>atin:Na ):1 with</td> <td>aCMC, SDS</td> <td>(3 g oil)/150 mL of gelatin with 150 mL of NaCMC</td> <td>Spray drying</td> <td>190 ± 3</td> <td>90 ± 3</td> <td>PV (2.98 mEq/Kg)</td> <td>20-0ct</td> <td>Food</td> <td>Spray drying (75.2) &amp; freeze- drying (53.2)</td> <td>atrick and others (2013)</td>	atin:Na ):1 with	aCMC, SDS	(3 g oil)/150 mL of gelatin with 150 mL of NaCMC	Spray drying	190 ± 3	90 ± 3	PV (2.98 mEq/Kg)	20-0ct	Food	Spray drying (75.2) & freeze- drying (53.2)	atrick and others (2013)
$ \begin{tabular}{ c c c c c c c c c c c c c c c c c c c$	P, WPC 30% WC 30% WC 30% WC 50% S(	r, WPI, PI with PC, & PI with	1:02	Freeze-drying Spray drying	NM 140, 160, & 180	Z Z	WN	1.37 to 4.59	Food	59.05 to 85.67	Aghbashlo and others (2013b)
<ul> <li>I:02 Spray drying 177.23 NM Reduced the susceptibility of fish oil for ovidative for ovid</li></ul>	P, 20%		1:02	Spray drying	140, 160, & 180	NM	NM	1.58 to 8.73	Food	59.05 to 85.67	Aghbashlo and others
WPI, Plwith     1:02     Spray drying     140:180     77.85:103.20     NM     1.37 to 4.59     Food     NM     Aghbashlo and others       PL, & PL, & Plwith     PL     1.37 to 4.59     Food     NM     Aghbashlo and others     (2012b)       Plwith     Spray drying (coupling RSM     220     NM     PV (5.6.6.7     2.31 to 5.48     Food     76.81 to 95.07     Aghbashlo and others       and genetic algorithm)     and genetic algorithm)     220     NM     PV (5.6.6.7     2.31 to 5.48     Food     76.81 to 95.07     Aghbashlo and others	P, 20%		1:02	Spray drying	177.23	MN	Reduced the susceptibility of fish oil for oxidative reactions	1.87 to 7.18	Food	79.14	Aghbashlo and others (2012c)
SM         NM         Spray drying         220         NM         PV (5.6.6.7)         2.31 to 5.48         Food         76.81 to 95.07         Aghbashlo and others           (coupling RSM         mEq/Kg)         mEq/Kg)         2.31 to 5.48         Food         76.81 to 95.07         Aghbashlo and others           and genetic         and genetic         (2012a)         (2012a)         (2012a)	P, WPC 30% W 30% N 30% N 30% S	C, WPI, PPI;with PPC, & /PI with	1:02	Spray drying	140:180	77.85:103.20	WN	1.37 to 4.59	Food	WN	Aghbashlo and others (2012b)
	P, by R lesign	SM	WZ	Spray drying (coupling RSM and genetic algorithm)	220	WN	PV (5.6:6.7 mEq/Kg)	2.31 to 5.48	Food	76.81 to 95.07	Aghbashlo and others (2012a)

Table 2-Overview of microencapsulated oils and their characteristics

Table 2–Continu	ied.									
				Tempera	ture∕pH					
							The particle			
Oil type	Wall material, concentration	Ratio of core & wall	Technique	Inlet (°C)	Outlet (°C)	Oxidative stabil- ity/retention	capsules/DS $(\mu m)$	Applications	Encapsulation efficiency (%)	References
Tilapia oil	Gelatin, 4.5 wt%, xanthan, 0.3 wt%, & sucrose, 13.2 wt%	~1:2.5	Spray drying	121	MZ	Lipid oxidation (1.73 mmol/kg)	WN	Food	06∼	Huang and others (2014)
Fish oil	Barley protein,	Oil/protein ratio	Spray drying	150	$60 \pm 5$	Low oxidative	5-Jan	Food	92.9 to 100.2	Wang and others
Menhaden fish oil	Soluble rice bran fiber, 1.1% & SC 10%	— Menhaden oil (6.7%)	Spray drying	180 ± 2	WN	Good oxidative stability	Aug-62	Food	57.3	Wan and others (2011a)
Fish oil, tributryin, & resveratrol	Caseinate, 5.3%, glucose, 5.3%, & modified resistant starch, 5.3%	WZ	Complex coacervation	WZ	WN	WN	DS (7 to 9 for modified resistant starch	Food	WN	Augustin and others (2011)
Fish oil & olive oil	Sugar beet pectin, 2% & dried glucose syrup, 13% & 20 5%	1:01	Spray drying	180	80	Low stability oxidative.	8-Jan	Food	06 <	Polavarapu and others (2011)
Fish oil	Soybean soluble polysaccha- ride:MD, 1.6.5	1:02	Spray granulation & fluid bed film coating	60:70	30:40:00	PV (3.98 mEq/kg)	300 to 700	Food	96.44 to 97.75	Anwar and others (2010)
Fish oil	Alginate & starch	337.5 g/225 g	Spray drying	150	80	Oxidative protection achieved	18.6 to 19.8	Food & pharma- ceutical	60.8 to 76.6	Tan and others (2009)
Fish oil	N-octenylsucc- inate- derivatized starch, 4.5% & glucose syrup, 275%	Oil content 18%	Spray drying	170	70	Hydroperoxide concentration (88 to 146 mmol/kg)/ 11% & 33% RH	DS (0.52 to 4.13)	Food	WN	Drusch and others (2006)
Fish oil	MC, 28.37; 27.88 & MD 14.18; 13.94	1:3 & 1:1.5	Spray drying	160 ± 2	65 ± 2	Not improved	27	Food	84.8 to 86.5	Kolanowski and others (2006)
Fish oil	Malt dextrin & HPMC	¥ Z	Simple coacervation followed by spray drying	200	100	Good stability when replacing malt dextrin by 40% with	WZ	Food	~93 to 99	Wu and others (2005)
Fish oil	HPMC, MC, & MD	01:01.5	Spray drying	$160 \pm 2$	$60 \pm 2$	R (98.5%).	8.0 to 25.0	Food	98.5	Kolanowski and
Fish oil	SC, 10% & Carbohydrate, 10%	1:02	Freeze-drying	40 then 20		Satisfactory oxidation stability	After freezing 0.35 to 2.01	Food	53.3 to 81.6	Heinzelmann and others (2000a); Heinzelmann and others (2000b)
Microalgal oil	SC, 5% & MD, 14.5%	1:02	Spray drying	185	95 ± 5	PV (0.31 to 2.78 mmol/kg)	MN	Food	$88.02 \pm 0.37$ to $98.35 \pm 0.20$	Bao and others (2011)
										(Continued)

				Tempera	ture/pH					
				-			The particle size of			
Oil type	Wall material, concentration	Ratio of core & wall	Technique	Inlet (°C)	Outlet (°C)	Oxidative stabil- ity/retention	capsules/DS (μm)	Applications	Encapsulation efficiency (%)	References
Microalgal oil	Gelatin solution (1%, w/w, 500 mL) & GA solution (1%, w/w, 500 mL)	Oil (20 g) with mixture	Complex coacervation	pH of the emulsion was adjusted to 4.0, the cross-linked microcapsules were dried at 60/2 h		WN	30.50 ± 0.42	Food & pharma- ceutical	WZ	Zhang and others (2012a)
Flaxseed oil	MS (Hi-Cap 100 <sup>TM</sup> )/AG/WPC, (4/0/1, w/w/w)	1:05	Spray drying	180	80 ± 5	Preventing oxidation by nD/AG/WPC, (4/0/1,	3.19 to 5.74	Food	70 to 91	Tontul and Topuz (2013)
Flaxseed oil	GA, GA/MD, & GA/MD/WPI	2:03	Spray drying	175 ± 5	75 ± 5	GA w.v.w) GA & ternary mixtures of GA, MD, & WPI presented the highest protection from	Oct-50	Food	06 <	Gallardo and others (2013)
Flaxseed oil	CPI or LPI & MD (25.0% to 40.7%)	MN	Freeze-drying	-40: -50		PV (5.76 to 6.40 mEq/kg)	D4.3 values of 3.7 & 3.9 (LPI- and CPI-stabilized emulsions),	Food	~ 83.5	Karaca and others (2013a)
Flaxseed oil	MD with GA, WPC or 2 types of MS (Hi-Cap 100 <sup>TM</sup> & Capsul TA®), 25:75	1:04	Spray drying	180 ± 2	110±2	The mixture of MD:WPC performed better in protecting the active material against oxidation	0.02 to 160.0	Food	62.3 to 95.7	Carneiro and others (2013)
Flaxseed oil	CPI or LPI & MD (20% oil, 20% protein, 60%	1:04	Benchtop spray dyer	180	90 ± 3	PV for CPI (6.68 to 7.31 mEq/kg) & LPI (6.62 to 6.86)	DS (~22.4)	Food	~88.0	Karaca and others (2013b)
Flaxseed oil	GA, WPC, & a modified starch	Oil concentra- tions (10%, 20%, 30%, & 40%)	Spray drying	180 ± 2	100 土 4	Lowest lipid oxidation with modified starch	0.24 to 180.0	Food	37 to 97	Tonon and others (2012)
Flaxseed oil	MD/GA, (25% & 30%)	3:02	Spray drying	140	95	OSI (3.78 ± 0.01 h)	17.6 to 23.1	Food	54.6 to 90.7	Rubilar and others
Flaxseed oil with polyphenolic fractions from murta leaves	GA, 15%	4:03	Spray drying	110:180	75:125	The lowest oxidative rancidity	WN	Food	54.55 ± 3.61	Rubilar and others (2012b)
										(Continued)

				,						
				l empera	iture/pH		i			
							The particle size of			
Oil type	Wall material, concentration	Ratio of core & wall	Technique	Inlet (°C)	Outlet (°C)	Oxidative stabil- ity/retention	capsules∕DS (μm)	Applications	Encapsulation efficiency (%)	References
Flaxseed oil	Zein	28:01:00;	Spray drying	135	55:60	WN	WZ	Food	$93.26 \pm 0.95$	Quispe-Condori and others (2011)
Flaxseed oil	GA	4:01 3:01	Freeze drying Spray drying	NM 138:202	N N N N	Lipid oxidation (0.017 to 0.106	Diameters varying from	Food	59.63 ± 0.36 51.5 to 92.0	Tonon and others (2011)
Flaxseed oil containing shrimp	SC, 10% & lactose, 10%	1:02	Pilot scale spray dryer	180 ± 2	81.81	Hydroperoxide content (2.64 mmol/kg oil).	6 to 100	Food	83.61 to 84.84	Pu and others (2011)
Flaxseed oil	Gelatin-GA, total biopolymer concentrations	1:01	Complex coacervation	50/pH 4		No oxidation during 25 d of storage at room	WN	Food	84%	Liu and others (2010)
Epoxidized linseed oil	PVP	ELO-to-PVP ratio (0.33, 0.91, &	Spray drying	150	90:100	NM	16	Powder coating	~85	Senatore and others (2010)
Olive oil	MD, CMC, & lecithin/SC & BHT	01:01.5	Freeze-drying	-80		Protein constituents extended the shelf- life/unditerable	WN	Agri-food	99.79 ± 0.51	Calvo and others (2012)
Olive oil	Gelatin & SA,	Oil (0.5 to 7.0 g)	Complex	60		0il released	MM	Food	62.54 to 89.37	Devi and others
Olive oil & caffeic acid (CA)	SA solution, 1.5%	50 mg oil∕g alginate solution	Co-extrusion	40		~40% to 50% PV <(18 mEq/kg)/30 d & CA protected the MUFAs &	N.	Food	60.6	(2012) Sun-Waterhouse and others (2011)
Olive oil	gelatin, GA, and	1:02	Spray drying	$165 \pm 5$	$80\pm 5$	OSI (25.16 to	NM	Food	33.43 to 52.98	Calvo and others
Soy oil	Protein isolates from Phaseolus legumes (kidney, red, & mung beans), (6	10:1	Spray drying	180	80	(Except kidney protein isolate case) of the 3 protein isolates were very	15-Feb	Food	45.4 to 57.9	Liu and others (2014)
Soy oil	g/ 100 mL) SPI, 2.78% to 12 50% w///	1:02	Spray drying	160, 180, or 200	$\sim 80$	роогег циал эгт Higher	15-Feb	Food	40.38 to 71.80	Tang and Li
Soybean oil (eugenol-rich	Clove extract:MD:GA, 1.4 8.2 4	01:07.2	Spray drying	150	86	Nonpro-oxidative activity	15-Jan	Food	65	Chatterjee and Bhattacharjee
Clove oil	Gelatin with sodium carboxymethyl guar gum	WN	Complex coacervation	40/(pH 2.5:4)		WN	Clove oil microcapsules (102) & sulfamethoxa- zole capsules (110)	Pharmaceutical	N N	Thimma and Tammishetti (2003)
										(Continued)

				Tempera	nture/pH					
							The particle			
Oil type	Wall material, concentration	Ratio of core & wall	Technique	Inlet (°C)	Outlet (°C)	Oxidative stabil- ity/retention	capsules/DS (μm)	Applications	Encapsulation efficiency (%)	References
Oregano oil	IN, 5%, 15%, & 25%	15% solids basis	Spray drying	120 & 190	WN	WN	3 to 4.5	Food	WN	Beirao da Costa and others
Oregano oil	Rice starch, 20% & Gelatin, 1%	0.4 to 3	Supercritical solvent impregnation	(40 to 50)∕ impre 24 h)	ignation time (3 to	High antioxidant activity	(<10)	Food	It has been evaluated by the antioxidant	Almeida and others (2013)
Oregano oil	Rice starch porous spheres, IN, & gelatin/sucrose	15% solids basis	Spray-drying & Freeze-drying	120:170	N Z	Gelatin/sucrose microparticles exhibit high	WN	Food	activity NM	Beirão da Costa and others (2012)
Oregano oil, citronella, & marjoram	SMP & WPC, 30% concentration (w/w)	2:03	Spray drying	190 ± 5	90 ± 5	NM	6 to 280 (SMP) & 2 to 556 (WPC)	Food	54.3 to 80.2	Baranauskienė and others (2006)
Sunflower oil	WPI or SC, 4% & trehalose, 13.6%	Oil phase 22%	Spray drying	150	60	PV (0.08 to 0.09 mEq/kg)	(10 to 70) while the agglomerates (120 to 300)	Food	96 (WPI), 99 (SC), & 90 to 96 after the agglomera-	Domian and others (2014)
Sunflower oil and astaxanthin	Soluble corn fiber with WPI or SC as emulsifiers	1:04	Spray drying	160, 170, & 180	70 & 80	~45% to 70%	25-Feb	Food	~95	Shen and Quek (2014)
Sunflower oil	MD, 6% & HPMC,	1:02	Spray drying	163	NM	NM	MM	Food	73.13 to 87.00	Roccia and
Sunflower oil & camelina	Dried glucose syrup, 889 or 15.71 & SC, 2.31	WN	Spray drying	180	80:82	Sunflower oil, emulsions, and reconstituted powders had lower oxidation than camelina oil/storage at	53 to 109	Food	Sunflower, 49.51 to 87.38 & Camelina, 49.84 to 61.65	or Dwyer and others (2013)
Sunflower oil	(FG)–(GA), a total biopolymer concentration lower than 4% w/w using weight ratios of FG to GA from A0-60 to 50-70	WN	Complex coacervation	рН 3.5		2 WZ	DS (40 to 240)	Food	WZ	Piacentini and others (2013)
Sunflower oil	MD, 20.0%	1:07	Nonfoamed & foamed spray drying	150:200	79:120	WN	Nonfoamed spray-drying (47.9 to 56.1) & foamed (52.5 to 98.3)	Food & pharma- ceutical	Nonfoamed spray-drying (87 to 65) & foamed (52 to 85)	Lewandowski and others (2012)
										(Continued)

Table 2–Continu	ied.									
				Tempera	ature∕pH					
Oil type	Wall material, concentration	Ratio of core & wall	Technique	Inlet (°C)	Outlet (°C)	Oxidative stabil- ity/retention	The particle size of capsules∕DS (µm)	Applications	Encapsulation efficiency (%)	References
Sunflower oil	HPMC, 0.7%; NaCMC, 0.3%; & SDS, 0.00%	20% w/w 0/W	Pre-, postcoacer- vation & coacervation	WN		WZ	WN	Food	WN	Katona and others (2010)
Cardamom oleoresin	Blends of GA, MD, & MS, & A/6,1/6,1/6	1:06	Spray drying	178 ± 2	120 ± 5	A 4/6,1/6,1/6 blend of GA:MD:MS offered a protection	N	Food	WZ	Krishnan and others (2005)
Cardamom oil	Mesquite ( <i>Prosopisjuli-</i> fora)	1:04	Spray drying	$200 \pm 5$	110±5		DS (2.59)	Food	83.6	Beristain and others (2001)
Black pepper oleoresin	gam GA & MS	1:40	Spray-drying	178±2	$110 \pm 5$	Higher for GA than for	Modified starch (5 to 15), GA	Food	WN	Shaikh and others (2006)
Cumin oleoresin	GA, MD, & MS, 4/6:1/6:1/6	1:03	Spray drying	$160 \pm 2$	$120 \pm 5$	Stability of volatiles	(/ [0 20]) NM	Food	Blends more efficient than	Kanakdande and others (2007)
Kenaf seed oil	SC:MD, 1:9	1:03	Spray drying	160	$85 \pm 2$	Resistant to lipid	DS(0.13)	Food	$97.02 \pm 0.52$	Ng and others
Kenaf seed oil	SC & MD	1:03	Spray drying	160	85 ± 2	Good oxidative	Distribution	Food	NM	Razmkhah and
Kenaf seed oil	SC, MD with soy lecithin	1:03	Spray drying	160	80 ± 2	The highest oxidative	25.64 to 45.75	Food	96.46	Vitters (2013) Ng and others (2013b)
Peppermint oil	Alginate-pectin mixture, 1.5 g/100 mL water	WN	Coaxial electrospray system	After electrospray, freeze-dried at $-120$ °C/ 1 d using a freeze-drier		Affected by the alginate: pectin ratio	1.58 to 3.24	Food	80.38 to 85.15	Koo and others (2014)
Mint oil	GGH modified with OSA & oleic acid & compared with (GA) & GA-OSA	20% w/v solution gum with 3 g (15% gum) of mint oil	Spray drying	160	95 ± 2	54.89% to 64.33%	(2.29 to 15.45) GA, (2.11 to 16.44) GGH-oleate, (2.56 to 15.56 to 15.56 to 15.22 to	Food, perfumery, flavoring, & pharmaceuti- cal	72.98 to 84.19	Sarkar and others (2013)
Mint oil	GA & radiation or enzymatically depolymerized guar gum	WN	Spray drying	160	N N	$(70.13 \pm 1.88)$ to (88.12 ± 1.57)	(2.12 to 15:13) (2.12 to 15:13) (2.47 to 16.11) GA:IRP, & (2.41 to 14.13) GA:IRS	Food, pharma- ceutical, & cosmetics	67.56 ± 2.62 to 881.27 ± 1.29	Sarkar and others (2012)
										(Continued)

				Tempera	iture∕pH					
							The particle size of			
Oil type	Wall material, concentration	Ratio of core & wall	Technique	Inlet (°C)	Outlet (°C)	Oxidative stabil- ity/retention	capsules/DS $(\mu m)$	Applications	Encapsulation efficiency (%)	References
Elemi and peppermint oils	MD, 30%	1:01	Spray drying	150	80	WN	WN	Food & pharma- ceutical	57.2 to 70.6	Adamiec and Kalemba
Peppermint oil	MD	10:1	Spray drying	150	80	Oil content in the powder after 5 mo 6.4 to 18.0 (wr%)	WN	Food	57.2 to 70.6	Adamiec (2009)
Peppermint oil	Gelatin, (2.5%, w/w, 400 mL)/ GA, (2.5%, w/ w, 400 mL)	1:02	Complex coacervation	190 ± 3	90 ± 3	Release about 7% of peppermint oil during the storage/40 d in	WN	Food & pharma- ceutical	× Z	Dong and others (2011)
Peppermint oil	OSA-modified starch		Spray drying	$200 \pm 10$	120±10	High retention	11.4 to 137.6	Food	88.8 to 99.7	Baranauskiene and others
Peppermint oil	Melamine & 37% formaldehyde	WN	<i>In situ</i> polymer- ization	$\sim$ 50 to 65		NN	At a rate of 13000 rpm	Functional fibers	87	(2007) Hwang and others (2006)
Rosemary oil	Starch:MD, (1:1) w/w	29.4:20.9% (w/w)	Spray drying	190	N N	R (9.1% to 54.3%)	12.2	Food	WN	de Barros Fernandes and others
Rosemary oil	Replacement of GA by MS, MD, & IN (1:1)	1:04	Spray drying	170	MZ	29.53 to 60.22	Starch (13.4), GA (13.5), & IN & MD (12.1)	Food	WN	Fernandes and others (2014)
Rosemary oil	GA, (10% to 30%)	1:04	Spray drying	171	NM	7.15% to 47.57%	13.6	Food	NN	Fernandes and
Walnut oil	SC, CMC, & MD	01:01.5	Freeze-drying	-80		CMC & MD with lecithin and BHT showed better oxidative stability	WZ	Food	36.90 to 69.09	(2011) (2011)
<i>Pterodon</i> oil	GA:MD, 3:3.6	1:(3:3.6)	Spray drying	130	06	R (70.55% β- caryophyllene∕ 45 d)	18.14 to 32.35	Medicinal & food	98.63	Alves and others (2014)
Garden cress seed oil	SC, WPC, blend of MD & GA & SMP	0.1-0.5 & 0.75	Spray drying	180 ± 2	90 ± 2	Good oxidative protection	13.3 to 31.3	Food	85.4, SACA>WPC > MDGA > SMP	Umesha and others (2013)
Thyme oil	Formaldehyde/ melamine molar ratio: 3.7	WN	<i>In situ</i> polymer- ization	70/(pH 5.0:5.2)		90%/4 wk	10-Jan	Food packaging & pesticide	22.6 to 77.5	Chung and others (2013)
Thyme oil	Polylactide solution in dimethyl formamide, 15.7 g/L	WN	Coacervation	WZ		WZ	40	Cosmetics	30.5	Martins and others (2009)
										(Continued)

				Tempera	ture/pH					
							The particle size of			
Oil type	Wall material, concentration	Ratio of core & wall	Technique	Inlet (°C)	Outlet (°C)	Oxidative stabil- ity/retention	capsules/DS (μm)	Applications	Encapsulation efficiency (%)	References
Clove, thyme, &	SA, 0.5, 1, 2, 4, or 8 w/ //06	1:03	Extrusion	NM		MM	MN	Pesticide	90 to 94	Soliman and
Macadamia oil	SC:MD, 1:4	2:03	Spray drying	167	WN	MN	7.5 to 10	Food	WN	Laohasongkram and others (2011)
Holy basil oil	Gelatin, 11.75% (w/v)	Ж <u>и</u>	Simple Coacervation	60∕ pH (3.5to 4.0)		Small decreases in (R) at 60 °C/ 49 d	392.3	Food, feed, medicinal, & pharmaceuti-	~95.5	Sutaphanit and Chitprasert (2014)
Citronella oil	Gelatin, 10% with sodium sulfate solution, 20%, & formaldehyde solution. 37%	1:1 & 2:1	Simple coacervation	50		WN	MN	Pharmaceutical & pesticide	60 to 70	Solomon and others (2012)
Green coffee oil	Modified starches (Hi-Cap, Capsul, and N-Lok) or GA with MD (75:25)	1:01	Spray drying	170	80:89	Mixture Hi-Cap/MD presented high oxidative stability	10.7 to 16.0	Food & cosmetics	82 to 99	Silva and others (2014)
Green coffee oil	Hi-Cap 100/corn syrup, 50:50 & Snow- flake/corn syrup, 50:50	WN	Spray drying	170	90 土 1	Only corn syrup showed lower oxidative stability	Lecithin- chitosan (14.51 to 19.50), lecithin (16.40 to 29.19)	Food & cosmetics	86 to 97	Carvalho and others (2013)
Coffee oil	GA	1:03	Spray drying	170	81:108	Stable during storage at 25 °C, but not at 60 °C	7.88 to 13.13	Food & cosmetics	48 to 82	Frascareli and others (2012)
Lippia sidoides oil	MD:GA ratio of 0:1 (m /m)	1:04	Spray drying	140, 150, & 160	NM	72%	8.35 to 15.87	Medicinal	NM	Fernandes and
Lemongrass oil	Poly(vinyl alcohol) (PVA, Polysciences	MZ	Simple coacervation	50		WN	10 to 250	Food & pharma- ceutical	MN	Leimann and others (2009)
Lemon myrtle oil	(MS & MD & WPC & MD)	1:1 & 1:2	Spray drying	200	80	54.53% to 90.07%	WN	food, cosmetics, and medications	71 to 86	Huynh and others (2008)
Lemon oil	eta-cyclodextrin	12:88 (w∕w)	Spray drying	160	60	R (~83)	NM	Food	$\sim$ 70 to 90	Bhandari and others (1999)
Zanthoxylum limonella oil	Chitosan, 2% (w/v) & gelatin, 2% (w/v)	WN	Complex coacervation	40⁄pH (5.4:5.9)		WN	WN	Mosquito repellent	32.47 to 60.05	Maji and Hussain (2009)
Zanthoxylum limonella oil	Gelatin, 4–10% (w/v) & glutaraldehyde, 25%	WN	Coacervation	40		WN	WN	Mosquito repellent	WN	Maji and others (2007)
										(Continued)

The particleThe particleThe particleThe particle113NMFor113NMNM113NMNM80 $\pm 2$ Microencapsu- lationHiCap (48), mologed the shelf-life from 1 visit $20\%$ RH), PV (memberyMD/GA(31) (20°C, mologed the mulsion 880 $\pm 2$ Microencapsu- lated oi above oins 20 mEq./kg) 8HiCap (48), (20°C, mologed the mulsion 880 $\pm 2$ Microencapsu- metoriapsu- oins 20 mEq./kg) 8HiCap (48), (20°C, mologed the mulsion 680 $\pm 2$ After 9 wk (20°C, mologed the oins 20 mEq./kg) 8For mulsion mulsion81 $\pm 5$ Covidation was slower when material NMNMFor mulsion81 $\pm 5$ Covidation was slower when material NM7.57For enulsion material NM80R (90% to 95%)26.71 to 47.52For to to some extent by microencap- sullationNMFor end extent by microencap- sullation80KGM-GA 69% & NMNMFor end extent by microencap- sullationNMFor end end end by microencap- NMNMFor end end end by microencap- sullationNMFor end end end by microencap- sullationNMFor end end end end end by microencap- sullationNMFor end e	
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				Tempera	ture∕ pH					
							The particle size of			
Oil type	Wall material, concentration	Ratio of core & wall	Technique	Inlet (°C)	Outlet (°C)	Oxidative stabil- ity⁄retention	capsules∕DS (μm)	Applications	Encapsulation efficiency (%)	References
Tea tree oil (TTO)	Gelatin solution (4–8% w/v) with olutaraldehvde	TTO (10:26 mL)	Simple coacervation	~ 40		WZ	WN	Medical purposes	52.95 to 87.57	Ocak and others (2011)
Evening primrose oil	Gd, MD, & Actor SC by RSM (17.2%, 75%, & 7.8%) respectively	WN	Spray drying	140	80 土 5	MD and/or GA mixed with a low amount of SC showed great improvement on oxidative	WN	Pharmaceutical & food	WN	Liu and Yang (2011)
Wheat germ oil	SA, 3% & starch, 0.5g	WN	The emulsification mothod	60/30 min		NM	84.2	Pharmaceutical	~ 88	Chan and others (2000)
Caraway oil	WPC/SMP & MD (9:1)	1:01	Spray drying	180±5	90 ± 5	Partial replacement of WPC by various MD enhanced protective against	WZ	Food	68.06 to 85.88	Bylaite and others (2001)
Vetiver oil	gelatin-GA	1:02	Complex	pH 4.0		NM	20 to 60	Food & pharma-	MM	Prata and others
Camphor oil	Gelatin with GA with added	oil⁄wall ratio, 0.75	Complex coacervation	рН 3.7		MN	85.7± 8.7:294.7± 14.2	Medicinal	99.6	Chang and others (2006)
Neem seed oil	NaCMC: gelatin, 1.0: 2.33	WN	Complex coacervation	$45 \pm 1/\text{pH}$ of 3.5		WN	NN	Pharmaceutical, medicinal, & nesticide	71.63 土 1.69 to 96.90 土 1.99	Devi and Maji (2011)
Neem seed oil	Chitosan, (0.5% w/v) & rx-carrageenan, (0.5% w/v)		Polyelectrolyte complex	45 & stirring/3 to 4 h to complete the cross-linking		WN	WN	Pesticide	66.02 to 98.47	Devi and Maji (2009)
Pomegranate seed oil	SMP	1:09	Spray drying	187	WN	MN	5.8 to 18.7	Food	95.6	Goula and Adamopoulos
Pimento oil	Chitosan & chitosan/ҡ- carraceanan	30:1, 40:1, & 67:1	Complex coacervation	25/pH 4.5		Chitosan improves the antioxidant activity	DS (4.87 to 6.35)	Food	92.16 to 98.31	Dima and others (2014)
Lavender oil	Gelatin, 1% & GA, 1%	3:02	Complex coacervation	25/pH 4		NM	40.5 to 139.6	Medicinal, perfumes, cosmetics	$66.0 \pm 0.3$	Xiao and others (2014)
Gac oil	(WPC 100)/GA:7/3, w/w	WN	Spray drying by RSM	150 ± 3	95 土 3	PV (3.91 mEq⁄kg)	<30	Food	73.59 to 96.67	Kha and others (2014b)
Schinus molle oil	MD & GA, 4:1 & 1-1	1:04	Spray drying	160	100	R (71%).	0.2 to 40	Pesticide	96 to 100	López and others
Vanilla oil	Chitosan, (1.0%, w/v) & GA, (2.0%, w/v)	2:01	Complex coacervation	рН 3		R (60%) in the microcapsules after release/30 d	5.2 to 10.3	Food	94.2	Yang and others (2014)
GA-HC, gum arabic-hen concentrate; SDS, sodiu butvl hvdroxytoluene: F	nicellulose; GA, gum arabic; H im dodecyl sulfate; MPC, milk G. fish gelatin: GGH, guar gur	HC, hemicellulose; WPI, whu protein concentrate; RSM m hvdrolvzate: OSA, n-octe	ey protein isolate; MD, mal , response surface method anvl succinic anhydride: KC	Itodextrin; IN, inulin; SC, Sc Jology; MC, methylcellulos	dium caseinate; AG, acac e; HPMC, hydroxypropyln 8-CD_8-cvclodextrin: DS	cia gum; NaCMC, sodium carbo nethyl cellulose; MS, modified dronlet size: NM, not mentio	oxymethyl cellulose; CMC, starch; CPI, chickpea prot ped: PV, peroxide value: O	sodium carboxymethyl cel ein isolate; LPI, lentil prot sl. ovidative stability inde	ulose; SMP, skim milk pow ein isolate; PVP, poly(N-vir z. RH_relative humidity. R	der; WPC, whey protein Jyl-2-pyrrolidone); BHT, retention

Encapsulation of marine, vegetable, essential oils ...



Figure 5-Schematic representation of the microencapsulation process by spray-drying.

An important step in developing microcapsules is the selection of a wall material that meets required criteria, including mechanical strength, compatibility with product, appropriate thermal or dissolution release, and appropriate particle size (Brazel 1999). The selection of wall materials for microencapsulation by spray-drying has traditionally involved trial-and-error procedures until microcapsules are formed. The capsules are then evaluated for several evaluations, for example, encapsulation efficiency, size particle, stability under different storage conditions degree of protection provided to the core material, and morphology of microcapsules by scanning microscopy (Perez-Alonso and others 2003).

The main reason that limit of the spray-drying technique in microencapsulation is the limited number of wall materials available and that must have a good solubility in water. Another disadvantage for spray-drying that should be considered is that it produces a fine microcapsule powder, which needs further processing such as agglomeration. In addition, lower oxidative stability due to the high temperatures used during the atomization process.

## Coaxial electrospray system

Coaxial electrospray system is a novel technology to encapsulate oils for the food, cosmetics, and pharmaceutical industries (Zhang and others 2011; Koo and others 2014). Over the past few years, single-fluid electrospraying has been explored in the preparation of drug-loaded systems with both sustained and rapid drug release profiles (Li and others 2014). Recently, a 2-phase coaxial electrospray system was used. Outer and inner solutions are coaxially and simultaneously sprayed through 2 separate feeding channels into one nozzle. In the electrospray process, the compound Taylor cone with a core–shell structure is formed on the top of the spray nozzle, and the outer polymeric solution encapsulates the inner liquid. The small charged droplets are formed when the bulk liquid is broken by coulomb repulsion (Figure 6). The most outstand-

ing features of the electrospray method are that it is easy, rapid, and efficient with maximum retention of the core material, that is, the maximum encapsulation efficiency (85.15%) was obtained with microencapsulation of peppermint oil in an alginate-pectin matrix (Table 2). It has been mentioned that encapsulation efficiency and the stability of the microcapsules were affected by wall materials (Koo and others 2014). Figure 6 shows a schematic exemplification of the 2-phase coaxial electrospray system, which is comprised of 2 syringe pumps, a stainless steel nozzle containing a needle, and a high-voltage generator. The wall liquid was prepared in a syringe pump (2) and the core liquid was injected using the syringe pump (1). The nozzle needle had an outer and inner diameter of 0.51 and 0.2 mm, respectively. Each syringe was attached to a programmable syringe pump. A voltage in the range of 0 to 30 kV and a limiting current of 2 mA generated by a high-voltage generator were applied to the coaxial nozzle (Koo and others 2014).

Coaxial electrospray has several advantages such as uniform size distribution, high encapsulation efficiency, and effective protection of bioactivity. Correspondingly, process control in coaxial electrospray is difficult to some extent (Zhang and others 2012b).

## Freeze-drying

Freeze-drying, also known as lyophilization or cryodesiccation, is a simple process as shown in Figure 7, and is used for the dehydration of almost all heat-sensitive materials and aromas like oils. Before drying, the oil is dissolved in water and frozen (between -90 °C and -40 °C; Heinzelmann and others 2000a,b) and then the surrounding pressure is reduced and enough heat is added to allow the frozen water in the material to sublimate directly from the solid phase to the gas phase (Oetjen and Haseley 2004). Freeze-dried materials seem to have the maximum retention of volatile compounds in comparison to that of Encapsulation of marine, vegetable, and essential oils ...



Figure 6–Schematic representation of the microencapsulation process by coaxial electrospray system.



Figure 7–Schematic diagram of a freeze-dryer.

spray-drying (Krokida and Philippopoulos 2006). This technique has been used successfully for microencapsulating some oils such as fish, flaxseed, walnut, and olive oil. Highest encapsulation yields  $(99.79 \pm 0.51\%)$  were achieved for olive oil when MD, CMC, and lecithin were used as encapsulants and the ratio of oil-wall material was 1:1.5 (Heinzelmann and others 2000b; Quispe-Condori and others 2011; Calvo and others 2012; Karaca and others 2013a). Besides protecting heat-sensitive core materials, freeze-drying is simple and easy to operate. It has been found that freeze-dried samples were more resistant to oxidation and lower microencapsulation efficiency (Velasco and others 2003). The major disadvantages include high energy use, long processing time, and high production costs compared to other drying methods (Desobry and others 1997). Freeze-dried ingredients may have higher porosity, thereby exposing the core material to the surrounding environment. However, the porous structures of freeze-dried bioactive products offer a higher drug release (Sinha and others 2007).

#### Coacervation

Coacervation is one of the oldest and most widely used encapsulation techniques. It involves the electrostatic attraction between 2 biopolymers of opposing charges, and coacervates formation occurs over a narrow pH range. In this technique, the liquid phase separates from the polymer-rich (coacervate) phase. The coacervation process is widely used in the food (Leimann and others 2009; Katona and others 2010; Liu and others 2010; Augustin and others 2011; Dong and others 2011; Xiao and others 2011; Devi and others 2012; Zhang and others 2012a; Piacentini and

others 2013; Tamjidi and others 2013; Sutaphanit and Chitprasert 2014; Yang and others 2014), pharmaceutical (Thimma and Tammishetti 2003; Chang and others 2006; Leimann and others 2009; Devi and Maji 2011; Dong and others 2011; Ocak and others 2011; Solomon and others 2012; Zhang and others 2012a; Sutaphanit and Chitprasert 2014; Xiao and others 2014), cosmetics (Maji and others 2007; Martins and others 2009; Xiao and others 2014), and pesticide industries (Maji and Hussain 2009; Devi and Maji 2011; Solomon and others 2012).

The coacervation process has been classified into simple and complex coacervation. Simple coacervation has been used to encapsulate fish, basil, citronella, lemongrass, and tea tree oil (Wu and others 2005; Devi and Maji 2009; Leimann and others 2009; Ocak and others 2011; Solomon and others 2012; Sutaphanit and Chitprasert 2014). In simple coacervation, the polymer is salted out by the action of electrolytes, such as sodium sulfate, or desolvated by the addition of a water-miscible nonsolvent, such as ethanol, or by increasing/decreasing the temperature. These conditions promote the macromolecule-macromolecule interactions (Martins and others 2009; Ocak and others 2011). It readily allows the production of microcapsules containing hydrophobic substances, such as of marine, vegetable, and essential oils. Simple coacervation offers important advantages over complex coacervation with regard to cost-saving and flexible operations. To induce the phase separation, simple coacervation uses inexpensive inorganic salts, whereas complex coacervation is more sensitive to even a small pH change. Furthermore, complex coacervation uses relatively expensive hydrocolloids (Sutaphanit and Chitprasert 2014).

Complex coacervation is a process in which 2 or more oppositely charged polymers are involved. This method has also been used to microencapsulate fish, microalgal, flaxseed, olive, clove, sunflower, peppermint, sweet orange, vetiver, camphor, neem seed, lavender, vanilla, and *Z. limonella* oils (Thimma and Tammishetti 2003; Chang and others 2006; Prata and others 2008; Devi and Maji 2009; Maji and Hussain 2009; Liu and others 2010; Augustin and others 2011; Dong and others 2011; Xiao and others 2011; Devi and others 2012; Zhang and others 2012a; Piacentini and others 2013; Tamjidi and others 2013; Wang and others 2014a; Yang and others 2014).

A schematic diagram of complex coacervation is shown in Figure 8. The 1st step in microencapsulation of oil by the complex coacervation process involves emulsification of the oil in an aqueous solution containing 2 different polymers (most commonly a polysaccharide and a protein), usually at a temperature and pH



Figure 8–Example of complex coacervation involving (i) emulsification of oil in an aqueous solution containing 2 different polymers, (ii) initial coacervation of polymers after lowering the solution pH below the isoelectric point of protein, (iii) deposition of the polymers on the surface of the oil, and (iv) wall hardening by addition of a cross-linking agent.

above the gelling and isoelectric point of protein. The 2nd step is the separation of the liquid phase from the insoluble polymer-rich phase as a result of the electrostatic attraction between oppositely charged polymers caused by lowering the solution pH below the isoelectric point of protein. The 3rd step consists of wall formation due to the deposition of the polymer-rich phase around the hydrophobic droplets, followed by controlled cooling below the gelling temperature. In the last step, wall hardening of microcapsules is achieved by the addition of cross-linking agents (Piacentini and others 2013).

Polyelectrolytes (macromolecules) carry a relatively large number of functional groups that either are charged or, under suitable conditions, can become charged (Du and others 2006). Polyelectrolyte complexes (PEC) are formed simultaneously by mixing oppositely charged polyelectrolytes in a solution without the addition of any chemical covalent or cross-linker that may induce toxicity. PEC is generally nontoxic, well-tolerated, and biocompatible. The major interactions between 2 polyelectrolytes include the strong, but reversible, electrostatic, and dipole-dipole association, and hydrogen bonding (Ii'ina and Varlamov 2005). Various neutral polymers, such as hydroxypropyl methylcellulose, cationic polymer (such as chitosan), and anionic polymers like  $\kappa$ -carrageenan and SA have been used in the formation of PEC such as SA-chitosan, polyacrylic acid-chitosan, and chitosan-carrageenan to design controlled release formulations, especially drugs. Neem (Azadirachta indica A. Juss.) seed essential oil, which acts as a natural liquid pesticide, has been successfully encapsulated using  $\kappa$ -carrageenan and chitosan polyelectrolyte systems (Devi and Maji 2009).

The wall-forming polymer plays an important role in the coacervation technique because it is responsible for the protection of the microencapsulated oil. PVA is a hydrophilic polymer that can be used as a wall-forming material in microcapsules by simple coacervation. PVA can be cross-linked with glutaraldehyde, thereby forming a hydrogel. PVA is also interesting because of its relatively simple chemical structure, ease of processing, and its potential use in pharmaceutical and biomedical fields (Leimann and others 2009). Nevertheless, for simple and complex coacervation, gelatin is the most frequently used microencapsulating

material (Prata and others 2008; Sutaphanit and Chitprasert 2014; Wang and others 2014a). The wall material systems which are most widely investigated include gelatin/gum (Thimma and Tammishetti 2003; Chang and others 2006; Prata and others 2008; Liu and others 2010; Dong and others 2011; Zhang and others 2012a; Piacentini and others 2013; Tamjidi and others 2013; Xiao and others 2014), gelatin/alginate (Devi and others 2012), gelatin/glutaraldehyde (Maji and others 2007; Ocak and others 2011), gelatin/chitosan (Maji and Hussain 2009; Yang and others 2014), and gelatin/CMC (Katona and others 2010; Devi and Maji 2011; Patrick and others 2013). However, gelatin is quite viscous even in low concentrations, therefore, other systems including caseinate/glucose/modified resistant starch (Augustin and others 2011) and plant proteins are also used (Ducel and others 2004; Xiao and others 2011).

The complex coacervation technique produces microcapsules with low surface oil and higher oil content and stability, as compared to that of spray-dried emulsions. Microcapsules produced by coacervation possess excellent controlled release characteristics and heat-resistant properties (Xiao and others 2011; Kralovec and others 2012; Wang and others 2014a). The major advantage of complex coacervation over other methods is that it has a very high payload (up to 99%). In addition, this method is simple, scalable, of low cost, solvent-free, and reproducible to obtain microencapsulated oils. Therefore, complex coacervation can be used for the fabrication of microcapsules on an industrial scale (Xiao and others 2014).

#### In situ polymerization

In situ polymerization has become the most commonly used method for the preparation of microcapsules and functional fibers. In situ polymerization results in the formation of a wall via the addition of a reactant into either the interior or the exterior of the core material (Hwang and others 2006). The distinguishing characteristic of in situ polymerization, rather than any other polymerization process for encapsulation, is that no reactants are included in the core material. All polymerization occurs in the continuous phase rather than on both sides of the interface between the core material and the continuous phase. The microcapsule formation process is conducted by the emulsion of oil in a melamineformaldehyde resin solution and a sonication process to emulsify the oil in the aqueous phase, then the resin is added under stirring and then adjustment of the emulsion pH to acidic and finally microcapsule shell formation. This process promotes the reaction of melamine with formaldehyde at the interface of oil droplets, producing a cross-linked film of melamine-formaldehyde polymer as a microcapsule shell.

Microcapsules of peppermint, thyme, and tea tree oils have been prepared by the *in situ* polymerization technique, using a melamine–formaldehyde system for fragrance, insect repellent, food packaging, and footwear applications. The peppermint oil containing melamine–formaldehyde resin microcapsules results in smooth surface morphologies, and is capable of preserving the encapsulated fragrant oils for a sufficient periods of time. Furthermore, thyme oil microcapsules having a smooth surface showed good thermal and sustained release properties (Hwang and others 2006; Chung and others 2013). Sanchez–Navarro and others (2011) demonstrated that the incorporation of tea tree oil microcapsules, prepared by *in situ* polymerization in leather as a natural antimicrobial agent for use in shoes, provided the concept of "active shoe." Encapsulation of marine, vegetable, and essential oils ...



Figure 9-Example of the in situ polymerization technique for the microencapsulation of essential oils.

A polymer-based microcapsule shell is considered good due to its high strength and stability (Sanchez-Navarro and others 2011). On the other hand, Long and others (2009) used a copolymer to prepare microcapsules with low formaldehyde-melamine molar ratios to avoid formaldehyde toxicity. Melamine–formaldehyde microcapsules were prepared using an *in situ* polymerization process (Figure 9). The melamine–formaldehyde precondensate and copolymer are 1st precross-linked in water (w; step 1); oil is added (step 2) to form an O/W emulsion by agitation (step 3); the temperature of the emulsion is then raised leading to polymerization of the precross-linked monomer and the copolymer to form a wall on the dispersed oil surface (step 4). It has been revealed that significant reduction in the formaldehyde levels is possible while only slightly reducing the mechanical properties and still maintaining the encapsulation efficiency of about 75%.

### Extrusion

Microencapsulation of oils by extrusion is not frequently used compared to spray-drying. Recently, extrusion techniques have been used to encapsulate some vegetable and essential oils, including olive, clove, thyme, and cinnamon oils, for the food and pesticide industries (Sun-Waterhouse and others 2011; Soliman and others 2013). It has been used almost exclusively for microencapsulation of oils in a carbohydrate matrix. Clove, thyme, and cinnamon oils were microencapsulated through the extrusion technique to reduce the evaporation rate, thereby increasing their antifungal activity (Soliman and others 2013). Generally, extrusion microencapsulation includes 3 processes: (i) melt injection, (ii) melt-extrusion, and (iii) centrifugal extrusion (coextrusion), as shown in Figure 10.

In the melt injection process, the core material is dispersed in molten carbohydrate, and is then pressed through one or more dies (orifices) into a bath of cold, dehydrating liquid such as isopropanol and liquid nitrogen. The wall material solidifies with liquid, forming an encapsulating matrix to entrap the core material. The granules are recovered by filtration or centrifugation [Figure 10(i)]. The residual solvent is removed by air-drying or vacuum-drying. A continuous screw-extrusion process is also used for microencapsulation of oil into a solid matrix. The melt-extrusion process is similar to that of melt-injection. The major differences are that

the latter is a vertical screwless process with surface-washed particles, while the former is a horizontal screw process, with particles that are not surface-washed. This process is similar to the one used for making expanded cereal snack products. Extruders used in melt-extrusion are a cylinder which contains thermomechanical mixers that consist of one or more screws, and double-screw extruders equipped with self-wipe screws are favored for encapsulation [Figure 10(ii)]. Usually describe the extrusion screws by length/diameter ranging between 20:1 and 40:1 (Zuidam and Heinrich 2010). It has been demonstrated that adding the core material to the plasticized carrier matrix at a later stage of the screw-extrusion process protects sensitive bioactives (PUFAs) from the harsh extrusion conditions. The oxidative stability of microencapsulated oils can be improved by admixing acidic antioxidants (for example, citric acid, caffeic acid, ascorbic acid, or erythorbic acid) to the matrix to prevent contact of oxygen with the oil (Drusch and Mannino 2009; Sun-Waterhouse and others 2011). Centrifugal extrusion (coextrusion) is another special type of extrusion that consists of a concentric feed tube through which wall and core materials are pumped separately to the many nozzles mounted on the outer surface of the device (Desai and Jin Park 2005). While the core material flows through the center tube, wall material flows through the outer tube [Figure 10(iii)]. The coextrusion method was used in the encapsulation of olive oil using alginate microspheres (Sun-Waterhouse and others 2011). Results showed that oil encapsulated through the coextrusion technique was found more stable during storage.

The principal advantage of extrusion microencapsulation of oils is the stability of the oils against oxidation, low surface oil, and a prolonged shelf-life compared to that of spray-dried essential oils (Gouin 2004). Moreover, this technique helps to reduce the evaporation rate of essential oils (Soliman and others 2013); however, this process is more expensive (double the cost) than spraydrying. Another drawback is the rather large particles (from 150 to 2000  $\mu$ m), which limit the use of extruded essential oils in various applications (Desai and Jin Park 2005).

#### Supercritical fluid technology

Microencapsulation of essential oils through the use of supercritical fluid technology has much relevance for the

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Figure 10-Schematic diagram of microencapsulation by (i) melt injection, (ii) melt extrusion, and (iii) centrifugal extrusion (coextrusion) process.



Figure 11-Schematic diagram to show supercritical fluid technology.

pharmaceutical, cosmetic, and food industries and, in particular, has several inherent advantages: nontoxicity, easy removal of the solvent, no degradation of the product, and the process utilizes a large variety of materials producing controlled particle sizes and morphologies. Supercritical fluids are highly compacted gases that possess several advantageous properties of both gases and liquids. The used methods are gas antisolvent precipitation, particles from gas-saturated solutions, fluid extraction of emulsions, rapid expansion of supercritical solutions (Cocero and others 2009; Jyothi and others 2010). Recently, the supercritical solvent impregnation process that has proven to be successful for a wide variety of substances

(essential oils, fragrances, active pharmaceutical compounds, and dyes) and matrices (wood, polymers, cotton, and contact lenses). Essential oils can be degraded by the high processing temperatures used for spray-drying (above 80 °C) and the presence of oxygen, since the normal drying gas is compressed air. Alternatively, supercritical solvent impregnation was called "milder process," which is an environmentally friendly process where supercritical carbon dioxide is used as a green solvent. The apparatus used for the supercritical impregnation is schematically represented in Figure 11 and was adapted from Almeida and others (2013). This apparatus is operated in a batch mode and consist of a high-pressure stainless steel impregnation vessel, a temperature-controlled bath, a magnetic stirring plate, a pressure transducer, and a high-pressure carbon dioxide liquid pump. The impregnation cell is fed with a fixed amount of essential oils at the bottom. Wall materials are placed in a stainless mesh elevated from the bottom by a support. After loading the oils and the wall materials, the impregnation cell is immersed in the water bath at low temperature of less than 80 °C and carbon dioxide is then fed into the cell until the desired pressure is achieved in order to ensure the solubilization of active ingredients in supercritical carbon dioxide. It was demonstrated that encapsulation of oregano essential oil, with different types of starch by using supercritical solvent impregnation with supercritical carbon dioxide at 10 MPa, 40 °C, 6 h of impregnation, a mass ratio of 1.5, and low depressurization rate to preserve the antioxidant activity, avoided oil degradation, and ensured high diffusivity of supercritical carbon dioxide in the solid matrix (starch; Almeida and others 2013).

## Fluidized-bed-coating

Fluidized-bed-coating is one of the most efficient coating methods, which is finding ever-growing applications in the food and pharmaceutical industries. In this process, ingredients can be mixed, granulated, and dried in the same vessel, thereby reducing the material handling and processing times compared to other wetgranulation processes. Recently, the fluidized-bed-coating method was used to encapsulate fish oil by spraying it into the fluidized bed chamber followed by film-coating the granules (Anwar and Kunz 2011). Fluidized-bed-coating has other terms such as "air suspension coating" or "spray coating." It is accomplished by suspending solid particles of the core material by an air stream under controlled temperature and humidity, and then spraving the coating material. With time, the wall material will gradually build as a thin layer on the surface of the suspended particles. The coating material must have an acceptable viscosity to enable atomizing and pumping, should be able to form an appropriate film over a particle's surface, and must be thermally stable (Teunou and Poncelet 2005). The amount of material that coats the particles is dependent on the length of time that the particles are in the chamber; typically 5% to 50% of coating is applied. The various methods of fluid-bed coaters include top spray, bottom spray, and tangential spray is shown in Figure 12. In the top spray system, the coating solution is sprayed counter-currently downwards with air on to the fluid bed such that as the solid or porous particles move to the coating region they become microencapsulated. The opposing flows of the coating materials and the particles lead to increased encapsulation efficiency and the prevention of cluster formation. The top spray system has successfully been used to coat materials to get very small microcapsules ranging between 2 and 100  $\mu$ m. Top spray fluid-bed coaters produce higher yields of microencapsulated particles than either bottom or tangential sprays fluidized-bed coaters. The bottom spray, known as the Wurster system, is widely used for coating particles as small as  $100 \ \mu m$ . It uses a coating chamber that has a cylindrical stainless steel nozzle and a cribriform bottom plate. The cylindrical stainless steel nozzle is used for spraying the coating material during process. As the particles move from bottom to top through the cribriform bottom plate and pass the nozzle zone, they are microencapsulated by the coating material, which adheres to the microcapsule's surface by evaporation of the solvent or cooling of the microencapsulated particle. This process is continued until the desired thickness and weight is obtained. The multilayer coating procedure helps in decreasing particle defects, although it is a time-consuming process. The tangential spray

consists of a coating chamber with a rotatable bottom the same diameter as the chamber. During the process, the drum is raised to create a gap between the edge of the chamber and the drum. The tangential nozzle is placed above the rotating drum through which the coating material is released. Then, the particles move through the gap into the spraying zone and are encapsulated (Desai and Jin Park 2005). We can briefly summarize that, during processing of the tangential spray system, there are 3 mechanical forces which cause particle movement, mixing, and granulation. These are the centrifugal force (generated by the spinning of drum), lifting force (generated by the process air volume that passes through the adaptable drum gap), and gravity. These forces, resembling a spiraling helix, provide good mixing and result in the formation of particles with good content symmetry. Ideally, the particles to be coated by the fluid bed should be spherical and dense, and they should have a narrow particle size distribution and perfect flowability. Nonspherical particles have the biggest possible surface area and require more coating material for the same shell thickness than spherical ones, while if the edges are sharp they will damage the coating during handling. Moreover, because of the presence of the filter bags in the upper part of the machine, it is probably that the low-dense and fine particles accumulate of these filters. If necessary, more than one coating can be applied on the powder (Teunou and Poncelet 2005).

# **Applications of Microencapsulated Oils**

Due to a wide range of marine, vegetable, and essential oil applications interest is growing to encapsulate such oils to fully tap their potential benefits. Microencapsulated oils have found various applications in the fields of foods, pesticides, textiles, and pharmaceuticals (Table 3).

#### Food applications

Consumer interest has significantly increased in foods fortified by  $\omega$ -3 fatty acids. Such kind of foods are now readily available around the world, such as dairy products (cheese, yogurt, and milk), bread, spaghetti, pasta, juice, meat, and baby food products. Nevertheless, the challenge in producing fortified foods has been tremendous. The major challenge in producing these foods is related to the stability of oil in the product.

Marine oil is one of the most encapsulated oils that have been applied in food products, followed by some vegetable oils such as flaxseed oil. Increasing the amount of fish and flaxseed oils in human diets has become a popular trend in recent years due to a number of health benefits attributed to the consumption of  $\omega$ -3 fatty acids. Fish oil is a predominant dietary source of long-chain  $\omega$ -3 PUFA, as mentioned before. However, it is well-known that many lipids are sensitive to light, heat, and oxygen, and thereby undergo oxidative damage very quickly. Fatty acid oxidation is a major cause of food deterioration and can affect the texture, flavor, aroma, color, and shelf-life of food, which limits the use of fish oil for food fortification. Studies carried out by Ye and others (2009) demonstrated that microencapsulation of fish oil and incorporating it into processed cheese during processing results in less oxidation of long-chain  $\omega$ -3 PUFA, and the rheological properties of the processed cheese are not altered and maintained a higher sensory quality versus "fishy" off-flavor (Ye and others 2009). A similar study on the processed cheese also found that encapsulation of fish oils improved the oxidative stability and marketability of cheese fortified with fish oil (Rouse and others 2012), thus suggesting that an encapsulated fish oil emulsion is a useful carrier of  $\omega$ -3 LC PUFA for fortifying processed cheeses (Ye and

Microencapsulated oil	Product	Encapsulation efficiency/ retention (R)	Purpose	References
Flaxseed oil with vitamins E, A, and	Cheese	R (90% to 93%)	Functional foods	Stratulat and others (2014)
Fish oil Fish oil Fish oil	Processed cheese Processed cheese Queso fresco, cheddar, and mozzarella	NM NM The highest <i>w</i> -3 retention 8.69 mg/g	Functional foods Functional foods Functional foods	Rouse and others (2012) Ye and others (2009) Bermudez-Aguirre and
Fish & flaxseed oil	cneese Queso fresco, cheddar, and mozzarella	(cneadar) Better retention (8.49 mg∕g) in Outero freecro	Functional foods	Barbosa-Carlovas (2011) Bermudez-Aguirre and Barhosa-Canovas (2012)
Fish oil	Milk & yogurt	92.9% to 100.2%	Functional foods	Wang and others (2011)
Fish oil Tuna oil	Yogurt Orange juice, yogurt, or cereal bar	/ 5.49 ± 2.66% R (60.3% to 78.6%)	Functional foods	Estrada and others (2011) Shen and others (2011)
Soybean oil Fish oil	Yogurt Yogurt	MM MM	Functional foods Functional foods	Chen and others (2010) Tamiidi and others (2012)
"Palm olein" Structured lipids (SLs) containing long-chain PUFAs	Infant formula	%06	Functional foods	Nagachinta and Akoh (2013)
Fish oil Menhaden oil	Infant formula Baby food	NM 29%	Functional foods Functional foods	Curtis and others (2008) Wan and others (2011b)
Red salmon oil	Baby food	47.80%	Functional foods	Wan and others (2012)
Garden cress seed oil	Biscuits	64.80% 25 50% +0 01 40%	Functional foods	Umesha and others (2015)
Linseed on Marine oil	breau Spaghetti pasta	Z3:370 UG 31:470 NM	Functional foods	Verardo and others (2009)
Fish oil	Fruit juice	78.88 ± 2.89%	Functional foods	Ilyasoglu and El (2014)
Fish oil Fish oil & flaxseed oil	Dutch-style fermented sausages Dutch-style fermented sausages	MN MN	Functional foods Functional foods	Josquin and others (2012) Pelser and others (2007)
Olive oil with lemon juice	An instant sauce salad	NM E 4 E02 +- 00 702	Functional foods	Silva and others (2013)
Rosmarinus officinalis and Thymus	Mango fruit	04.0% 10 00.1% NM	Fungicides and pesticides without	Kupaei and Garmakhany (2014)
<i>vuigaris</i> oiis			negative errects on the environment and human health	
Thyme oil Cinnamon oil	Diets The films in packaging for cookies	90% 80.7%:94.6%	Insect repellent for food packaging Insect-resistant films for food	Chung and others (2013) Kim and others (2013)
Red pepper seed oil	Nonwoven fabric	MM	products Functional fabrics possessing	Ozyildiz and others (2013)
Clove bud oil & red thyme oil	Fabric	WZ	antimicrobial activity Reduce the population of house dust	Kim and Sharma (2011)
1 = 1 = - + 1 = 21 = 22 = - + 1 = - + - + - + - + - + - + - + - + - + -			mites (HDMs)	
Melaleuca alternitolia (tea tree) oll	Fabric used as footwear component	NM	Provide shoes with new properties and natural antimicrobial	Sanchez-Navarro and others (2011)
Citronella oil Lavender rocemary and sade oil	Cotton fabric	A repellent effect > 90% for 3 wk	Mosquito repellent Eracrant taxtila product	Specos and others (2010)
Lavenuer, rosennary, and sage on Lavender oil	Cotton fabric	WN	Fragrant textile product	Khajavi and others (2013)
Migrin oil	Cotton fabric	87% 67 306 to 81 406	Fragrant functional fabrics	Hong and Park (1999)
orange oil	Cotton fabric		Stability to water and detergents	Li and others (2013)
Berry oil	Cotton towel tabric	MM	Improve life quality of users by using multifunctional products with good	Sariisik and others (2012)
	- 11		anti-agnizing, relavation, anti-agni effects	
Limonene oli Jojoba oil Citronella oil	i extile Compressive knits Ointment	NM 92.22% 36.2% to 62.8%	rerrumea rextiles High burns "the medical uses" Mosquito repellent	Koorigues and others (2008) Jaafar and others (2012) Solomon and others (2012)
NM, not mentioned; R, retention.				

Encapsulation of marine, vegetable, and essential oils ...



Figure 12-Schematic diagram to show top, bottom, and tangential-spray fluidized-bed coating.

others 2009). However, flavor of cheese with added fish oil was preferred by panelists more than cheese fortified by encapsulated fish oil (Bermudez-Aguirre and Barbosa-Canovas 2011). It was observed that encapsulation of fish oil and incorporating it into other cheeses, such as queso fresco, cheddar, and mozzarella, improved the retention of  $\omega$ -3 into cheese (Bermudez-Aguirre and Barbosa-Canovas 2012). Recently, it was found that microencapsulation of  $\omega$ -3 fatty acid in cheese in the form of emulsified particles allowed increasing their retention in the curd, functional stability during storage, resistance of lipids to peroxidation, and better yield of fortified cheese (Stratulat and others 2014).

It has been found that microencapsulation of fish oil with barley protein and incorporating it into both milk and yogurt enhanced the oxidative stability during their storage (Wang and others 2011). Another research study found that the addition of encapsulated salmon oil to yogurt did not greatly modify pH, color, or waterholding capacity characteristics of strawberry yogurt (Estrada and others 2011). Tamjidi and others (2012) reported that the encapsulation of fish oil in complex coacervates of gelatin and acacia gum were found effective for the prevention of oxidation and rancidity of marine  $\omega$ -3 oil-enriched yogurt. In addition, fortified products showed low whey separation, high viscosity, and high water-holding capacity characteristics. It was suggested that it is possible to manufacture an enriched yogurt containing a high amount of fish oil powder and flavored for better sensory properties. An in vitro study with a dynamic artificial digestive system carried out by Chen and others (2010) demonstrated that yogurt fortified by microencapsulated soybean oil by SPI and zein complex significantly delayed the nutrient release (riboflavin), which could be desirable, because of the likelihood of reaching the bioactive components for intestinal absorption in an intact and active condition. In addition, the choice of food matrix may influence the lipolysis of microencapsulated oils during in vitro digestion. In another study, it was demonstrated that the lipolysis extent of microencapsulated tuna oil was low during in vitro digestion when it was delivered in a cereal bar (Shen and others 2011).

The application of commercial encapsulated fish oil seems to be the best in retaining overall quality of dry fermented sausages when enriched with n-3 fatty acids from fish oil. For example, it was found that the microencapsulated fish oil added into a dry fermented sausage had no clear effects in perceived bitterness, redness, spiciness, fishy taste, and fishy odor in addition to higher firmness

ratings and less advanced lipid oxidation (Josquin and others 2012). A physical and sensory analysis carried out by Pelser and others (2007) showed that the sausages containing encapsulated fish oil and flaxseed oil resembled the unfortified sausages.

The incorporation of encapsulated linseed oil in an optimized formulation of soup enhanced the oxidative stability and made it possible to provide a source of  $\omega$ -3 besides obtaining a value-added product with high consumer acceptability (Rubilar and others 2012a). Olive oil microcapsules prepared by freeze-drying were used successfully in instant salad sauce (Silva and others 2013).

The microencapsulation of fish oil can provide many benefits when incorporated into baby food products, such as providing an oxygen barrier resulting in an extended shelf-life, a taste profile barrier masking fish oil taste and odor, high nutrient density and availability, and a protective barrier against shear and temperature changes. Baby foods containing purified menhaden oil and encapsulated purified menhaden oil were evaluated and compared. It was found that unencapsulated menhaden oil had higher total  $\omega$ -3 PUFAs, total saturated, and total monounsaturated contents than microencapsulated menhaden oil when incorporated into baby food due to low encapsulation efficiency (29%; Wan and others 2011b). Investigations carried out by Wan and others (2012) revealed that encapsulated red salmon oil could be added to a commercial baby food product to increase the content of  $\omega$ -3 fatty acids while maintaining desirable attributes of the product. Curtis and others (2008) and Nagachinta and Akoh (2013) found that structured lipids containing long-chain PUFAs were successfully microencapsulated with high microencapsulation efficiency for infant formula applications.

Microencapsulation of marine oil in spaghetti was an effective means of protecting the fatty acids (long-chain PUFAs) from oxidation (Verardo and others 2009). Studies carried out by Gallardo and others (2013) on bread products demonstrated that the bread fortified with linseed oil microcapsules (made of 100% GA) had a similar appearance to that of the control bread. However, ALA content was reduced significantly after bread baking.

Recently, garden cress seed oil has been used as a source of PUFA because of its richness in ALA. Umesha and others (2015) found that microcapsules of garden cress seed oil added into biscuits offered oxidative protection to ALA during baking and increased the shelf-life of biscuits over long-term storage. Hence, microencapsulated garden cress seed oil can be incorporated and supplemented to increase ALA levels in biscuits or other food products. In another recent study, Ilyasoglu and El (2014) reported that fruit juice was successfully enriched with fish oil microencapsulated with stable protein–polysaccharide complexes.

#### Pesticide applications

Decay is a significant factor that limits the storage life of fruits and vegetables after harvest and is responsible for considerable economic losses. Fungicides and pesticides are commonly used to control the postharvest deterioration and losses in agricultural produce. However, due to harmful impacts of fungicides and pesticides on the environment and human health, and the development of fungicide resistance by pathogens, there is an urgent need to seek alternatives. Postharvest decay can be retarded by applying encapsulated essential oils, thereby avoiding the use of fungicides and pesticides. Recently, essential oils extracted from plants having repellent properties have been studied as replacements for chemical pesticides due to their environmentally friendly and biodegradable status. Studies carried out by Kupaei and Garmakhany (2014) demonstrated that postharvest treatment with encapsulated Rosmarinus officinalis and Thymus vulgaris essential oils to mango fruit had the potential to control decaying, prolong storage life, and maintain internal quality of the mango fruits. In recent years, insect-resistant packaging for food products has attracted attention from the food industry. Safety and quality of products can be ensured by using microencapsulated essential oils (green or environment-friendly insecticides) to replace chemical insecticides.

Chung and others (2013) found that diets containing thyme oil microcapsules possessed high insect-repellent efficacy (about 90%) for 4 wk. In another study (Kim and others 2013), films containing encapsulated cinnamon oil were prepared to protect food products from insects (*Plodia interpunctella*). It was found that the films containing cinnamon microcapsules were the most effective at repelling moth larvae and the release rate of cinnamaldehyde (an effective insecticide). In addition, tensile properties of the films were not changed and the invasion of larvae into cookies was shackled by the insect-repellent films, thus demonstrating the potential use of films in insect-resistant packaging for food products.

#### Textile and footwear applications

As pathogenic microorganisms are becoming more resistant to antibiotics, functional fabrics possessing antimicrobial activity have drawn considerable interest in recent years. Antimicrobial textiles have been widely used in home textiles and personal care products. Presently, disposable nonwoven textiles with antimicrobial properties are being used in the production of functional textiles. There are various methods to extend the antimicrobial properties of textiles including the use of microencapsulated antimicrobial agents within the fiber's matrix. Ozyildiz and others (2013) used microcapsules of ozonated red pepper seed oil, having antimicrobial properties, to nonwoven fabrics to manufacture disposable functional textiles. It was found that microcapsules containing ozonated red pepper seed oil were active against the test microorganisms. Functional fabrics possessing acaricidal activity have drawn significant interest to control insects including house dust mites (HDMs), which threaten human health by causing asthma, allergic rhinitis, and atopic dermatitis. The synthetic acaricides, such as pyrethroids, pose a risk of neurotoxicity to humans and other mammals. This problem warrants the development of safer, yet capable, alternative methods of controlling HDMs indoors. Clove bud and red thyme oil microcapsules, having acaricidal activities, were applied to fabric. It was noted that clove bud oil

microcapsules were more active at reducing the live HDMs (94% mortality; Kim and Sharma 2011). In another study carried out by Specos and others (2010), citronella essential oil microcapsules applied to cotton textiles had insect-repellent activities. Such fabrics demonstrated a higher repellent effect (>90%) and long (3 wk) lasting protection from insects compared to fabrics sprayed with bulk essential oil.

Microcapsules of *Melaleuca alternifolia* (tea tree) oil has been used as biocide in footwear applications. It was found that the encapsulation process was an effective method to protect these natural biocides from reactions with moisture, light, and oxygen. Moreover, *Melaleuca alternifolia* oil showed suitable antimicrobial activity against different microorganisms found in foot skin and worn shoes and, therefore, incorporation of such microcapsules in leather and fabrics have demonstrated the feasibility of this technology for use in shoes to achieve the concept of an "active shoe" (Sanchez-Navarro and others 2011).

The adsorption of perfumes and other fragrances onto solid carrier materials has been of interest to textile manufacturers because of their more convenient incorporation into textiles. However, volatile fragrances bound to solid carriers by adsorption are easily removed during ordinary washing processes with laundry detergents. It was found that the orange oil in the microcapsules was well retained in cotton fabrics after washing in a normal detergent solution and increased the persistence of fragrance on textiles (Li and others 2013). It has been found that microencapsulation of lavender, rosemary, and sage essential oils were used successfully to produce a fragrant textile product (Golja and others 2013). It was shown that encapsulation of lavender essential oil retained volatility of fragrance compounds on textiles, thus prolonging the fragrance of textiles (Khajavi and others 2013). Microcapsules of limonene oil were successfully incorporated into perfumed textiles (Rodrigues and others 2008). Moreover, microcapsules containing floral oil were synthesized and applied successfully into cotton fabrics (Lee and others 2002). In another study, migrin oil microcapsules applied on cotton fabrics were capable of preserving the oil fragrance for a long period of time (Hong and Park 1999).

## Pharmaceutical applications

There is a risk from mosquito-borne diseases in tropical areas, including dengue hemorrhagic fever, malaria, and filariasis. Solomon and others (2012) successfully used citronella essential oil microcapsules as mosquito repellents with ointment to the skin. It was found that microencapsulation reduced the rate of evaporation of the oil and offered a promising option to prolong the duration of action of citronella oil as a potential mosquito repellent.

Medical use of textile fibers is increasing day by day. For medical use, such materials should offer desirable characteristics, such as flexibility, softness, strength, elasticity, biostability, absorbency, and the ability to be sterilized. It has been found that the application of jojoba oil microcapsules onto compressive knits, developed for severe burns, preserved the initial characteristics of the knit, such as touch, flexibility, and lightness, besides playing a role in skin hydration and avoiding sebum accumulation (Jaafar and others 2012). In another study, a functional textile product with aromatic oil, having good odor, moisturizing, relaxation, and anti-aging effects, was designed for use at aromatherapy and spa centers or personal care to improve the quality of life for the users. Microcapsules of berry oil have also been successfully used for these purposes in 100% cotton towel fabrics (Sariisik and others 2012).

## **Conclusion and Future Prospects**

By the virtue of their biological, functional, and physicochem ical properties, marine, vegetable, and essential oils are being used in the preparation of safe products with a positive impact on consumer health. Nevertheless, they are chemically unstable and susceptible to degradation. Microencapsulation is an effective and important tool to prepare oil-based high-quality and health-beneficial products in various industries in order to enhance their chemical, oxidative, and thermal stability. Concomitantly, the shelf-life, biological activity, functional activity, controlled release. physicochemical properties, and overall quality of oils can also be enhanced. Spray-drying and coacervation are the most commonly used techniques for the encapsulation of oils. Microencapsulated oils have been successfully applied in various food, pharmaceutical textile, and pesticide products. Future research must be directed toward the use of microencapsulation technology to encapsulate a mixture of different oils by varying techniques. In this way, the oils' off-flavor can be masked and safety, quality, and the nutritional value of the product can also be improved. In addition, more than one microencapsulation technique can be utilized in the process. Other future suggestions include the purification and use of factory waste as a wall material, thereby reducing the economic cost of the encapsulation process. Given the near future in food products, it can be applied as antimicrobial edible films and coatings containing a microencapsulated oil, antimicrobial agent, and prolong the shelf-life of food products, thus furthermore decreasing the economic losses, especially cheeses products that are susceptible to fungal attack during ripening and storage. In medical products, it can be applied in adhesive bandages to control the occurrence of infections and in wheelchair patients to avoid skin infections.

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## **Conflict of Interest**

The authors declare no conflict of interest.

## Nomenclature

Acacia gum
$\alpha$ -Linolenic acid
$\beta$ -Cyclodextrin
Butylated hydroxytoluene
Carboxymethylcellulose
Chickpea protein isolate
Docosahexaenoic acid
Droplet size
Eicosapentaenoic acid
Fish gelatin
Gum arabic
Gum arabic-hemicellulose
Guar gum hydrolyzate
Hemicellulose
Hydroxypropyl methylcellulose
Inulin
Konjac glucomannan
Lentil protein isolate
Methylcellulose

	MD	Maltodextrin
_	MPC	Milk protein concentrate
z	MS	Modified starch
t	MUFA	Monounsaturated fatty acid
e	NaCMC	Sodium carboxymethylcellulose
_	NM	Not mentioned
ł	OSA	N-octenyl succinic anhydride
e	OSI	Oxidative stability index
e	PV	Peroxide value
,	PVP	Poly(N-vinyl-2-pyrrolidone)
e	RSM	Response surface methodology
y	SA	Sodium alginate
1	SC	Sodium caseinate
,	SDS	Sodium dodecyl sulfate
1	SMP	Skim milk powder
e	SPF	Sun protection factor
e	SPI	Soy protein isolate
_	WPC	Whey protein concentrate
,	WPI	Whey protein isolate
h	$\omega$ -3 PUFAs	Omega-3 polyunsaturated fatty acids

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